

PTO/PCT 07 JUL 2000

For receiving Office use only

REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference (if desired) (12 characters maximum) HMJ03045WO

Box No. I TITLE OF INVENTION
GYPSUM PRODUCT

Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

MOBIL OIL COMPANY LIMITED
Mobil House
500-600 Witan Gate
Central Milton Keynes
Buckinghamshire
MK9 1ES
United Kingdom

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State (that is, country) of nationality: GB

State (that is, country) of residence: GB

This person is applicant for the purposes of: ☐ all designated States ☒ all designated States except the United States of America ☐ the United States of America only ☐ the States indicated in the Supplemental Box

Box No. III FURTHER APPLICANTS AND/OR (FURTHER) INVENTORS

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

BOELEE, Scotia
105 Forest Road
Tunbridge Wells
Kent
TN2 5BG
United Kingdom

This person is:

☐ applicant only

☒ applicant and inventor

☐ inventor only (if this check-box is marked, do not fill in below.)

State (that is, country) of nationality: NZ

State (that is, country) of residence: GB

This person is applicant for the purposes of: ☐ all designated States ☐ all designated States except the United States of America ☒ the United States of America only ☐ the States indicated in the Supplemental Box

☐ Further applicants and/or (further) inventors are indicated on a continuation sheet.

Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: ☒ agent ☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

Gill Jennings & Every
Broadgate House
7 Eldon Street
London
EC2M 7LH
United Kingdom

Telephone No.

+44 171 377 1377

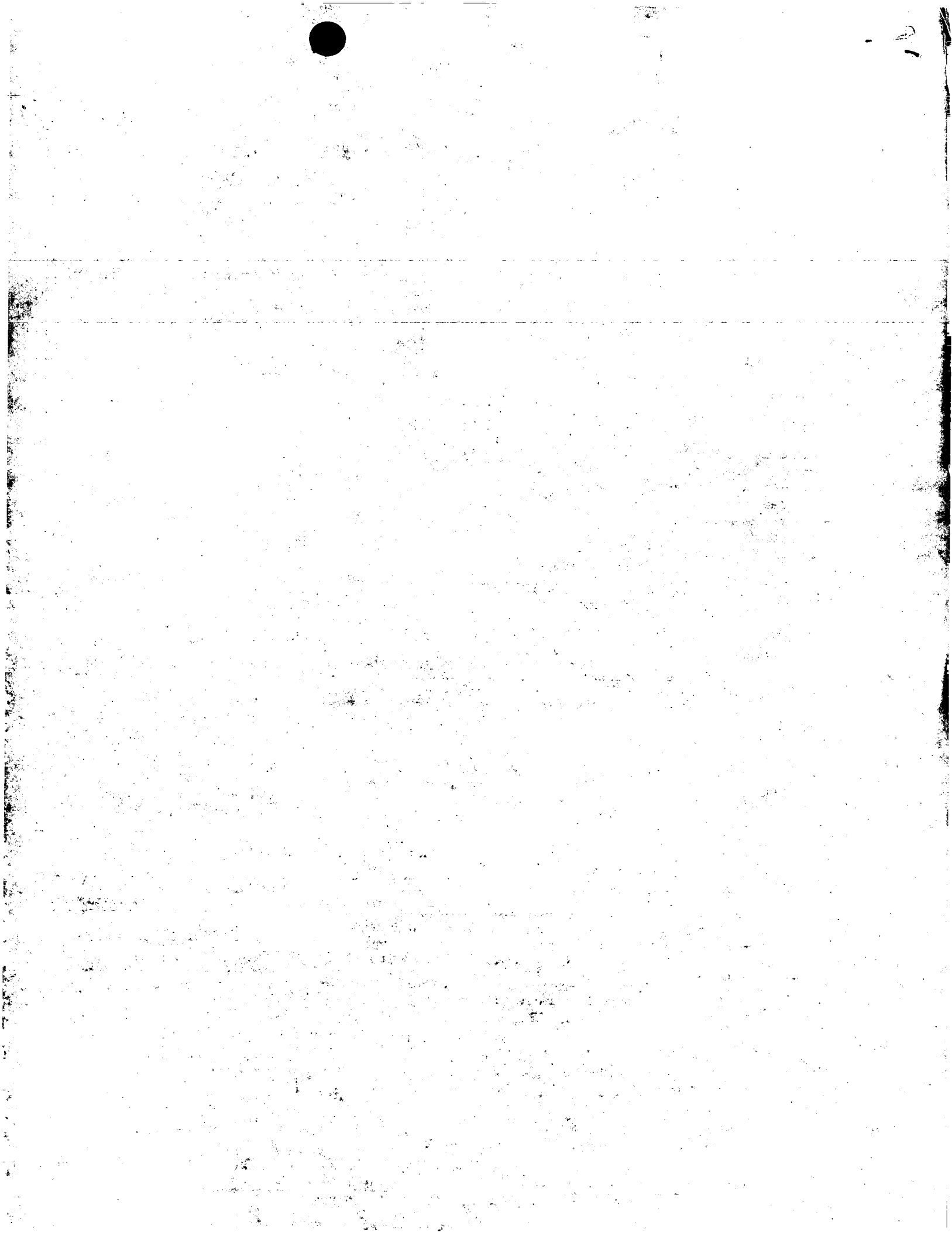
Facsimile No.

+44 171 377 1310

Teleprinter No.

(051) 22765 GILPAT G

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.



Box No.V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes; at least one must be marked):

Regional Patent

- ☒ AP ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe, and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ EA Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ EP European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ OA OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of the PCT (if other kind of protection or treatment is desired, specify on dotted line)

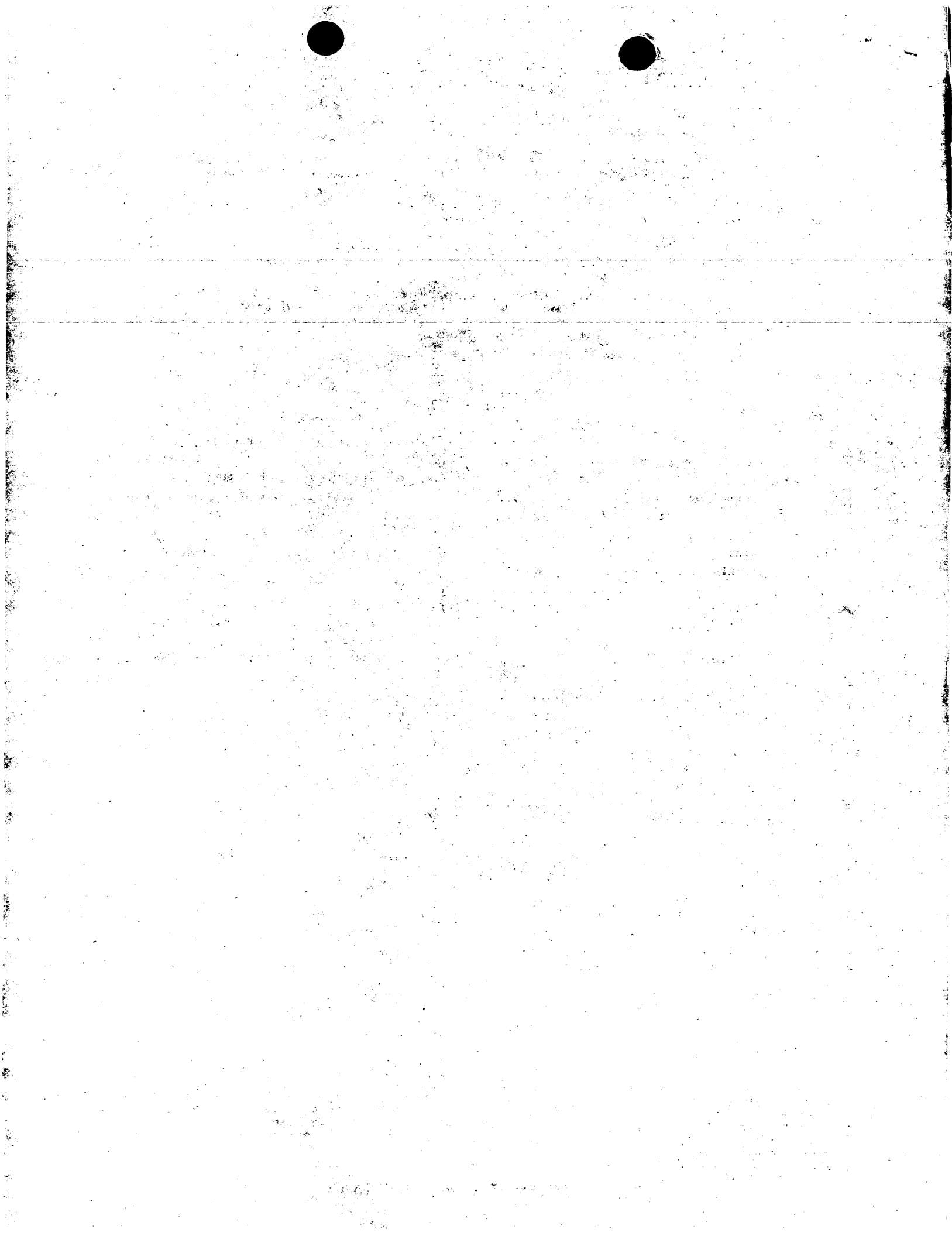
National Patent (if other kind of protection or treatment is desired, specify on dotted line):

- | | |
|--|--|
| <input checked="" type="checkbox"/> AL Albania | <input checked="" type="checkbox"/> LS Lesotho |
| <input checked="" type="checkbox"/> AM Armenia | <input checked="" type="checkbox"/> LT Lithuania |
| <input checked="" type="checkbox"/> AT Austria | <input checked="" type="checkbox"/> LU Luxembourg |
| <input checked="" type="checkbox"/> AU Australia | <input checked="" type="checkbox"/> LV Latvia |
| <input checked="" type="checkbox"/> AZ Azerbaijan | <input checked="" type="checkbox"/> MD Republic of Moldova |
| <input checked="" type="checkbox"/> BA Bosnia & Herzegovina | <input checked="" type="checkbox"/> MG Madagascar |
| <input checked="" type="checkbox"/> BB Barbados | <input checked="" type="checkbox"/> MK The former Yugoslav Republic of Macedonia |
| <input checked="" type="checkbox"/> BG Bulgaria | <input checked="" type="checkbox"/> MN Mongolia |
| <input checked="" type="checkbox"/> BR Brazil | <input checked="" type="checkbox"/> MW Malawi |
| <input checked="" type="checkbox"/> BY Belarus | <input checked="" type="checkbox"/> MX Mexico |
| <input checked="" type="checkbox"/> CA Canada | <input checked="" type="checkbox"/> NO Norway |
| <input checked="" type="checkbox"/> CH and LI Switzerland and Liechtenstein | <input checked="" type="checkbox"/> NZ New Zealand |
| <input checked="" type="checkbox"/> CN China | <input checked="" type="checkbox"/> PL Poland |
| <input checked="" type="checkbox"/> CU Cuba | <input checked="" type="checkbox"/> PT Portugal |
| <input checked="" type="checkbox"/> CZ Czech Republic | <input checked="" type="checkbox"/> RO Romania |
| <input checked="" type="checkbox"/> DE Germany | <input checked="" type="checkbox"/> RU Russian Federation |
| <input checked="" type="checkbox"/> DK Denmark | <input checked="" type="checkbox"/> SD Sudan |
| <input checked="" type="checkbox"/> EE Estonia | <input checked="" type="checkbox"/> SE Sweden |
| <input checked="" type="checkbox"/> ES Spain | <input checked="" type="checkbox"/> SG Singapore |
| <input checked="" type="checkbox"/> FI Finland | <input checked="" type="checkbox"/> SI Slovenia |
| <input checked="" type="checkbox"/> GB United Kingdom | <input checked="" type="checkbox"/> SK Slovakia |
| <input checked="" type="checkbox"/> GE Georgia | <input checked="" type="checkbox"/> SL Sierre Leone |
| <input checked="" type="checkbox"/> GH Ghana | <input checked="" type="checkbox"/> TJ Tajikistan |
| <input checked="" type="checkbox"/> GM Gambia | <input checked="" type="checkbox"/> TM Turkmenistan |
| <input type="checkbox"/> GW Guinea-Bissau | <input checked="" type="checkbox"/> TR Turkey |
| <input checked="" type="checkbox"/> HR Croatia | <input checked="" type="checkbox"/> TT Trinidad and Tobago |
| <input checked="" type="checkbox"/> HU Hungary | <input checked="" type="checkbox"/> UA Ukraine |
| <input checked="" type="checkbox"/> ID Indonesia | <input checked="" type="checkbox"/> UG Uganda |
| <input checked="" type="checkbox"/> IL Israel | <input checked="" type="checkbox"/> US United States of America |
| <input checked="" type="checkbox"/> IS Iceland | <input checked="" type="checkbox"/> UZ Uzbekistan |
| <input checked="" type="checkbox"/> JP Japan | <input checked="" type="checkbox"/> VN Viet Nam |
| <input checked="" type="checkbox"/> KE Kenya | <input checked="" type="checkbox"/> YU Yugoslavia |
| <input checked="" type="checkbox"/> KG Kyrgyzstan | <input checked="" type="checkbox"/> ZW Zimbabwe |
| <input checked="" type="checkbox"/> KP Democratic People's Republic of Korea | |
| <input checked="" type="checkbox"/> KR Republic of Korea | |
| <input checked="" type="checkbox"/> KZ Kazakhstan | |
| <input checked="" type="checkbox"/> LC Saint Lucia | |
| <input checked="" type="checkbox"/> LK Sri Lanka | |
| <input checked="" type="checkbox"/> LR Liberia | |

Check-boxes reserved for designating States (for the purposes of a national patent) which have become party to the PCT after issuance of this sheet:

- ☒ GD...Grenada.....
- ☒ IN...India.....

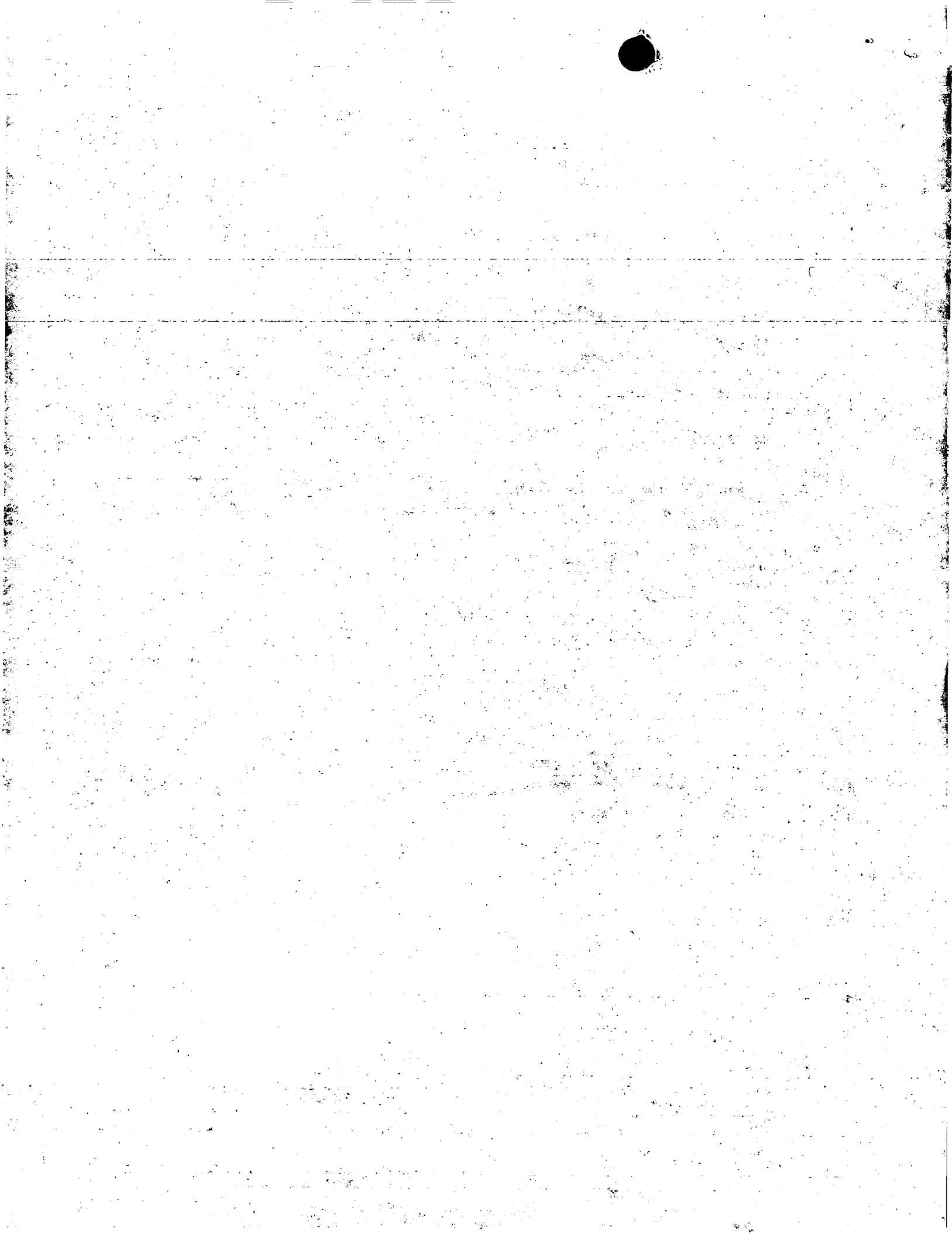
Precautionary Designation Statement: In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.)



Box No. VI PRIORITY CLAIM		<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.		
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application:* regional Office	international application: receiving Office
item (1) 08/01/1998 08 January 1998	9800368.4	GB		
item (2)				
item (3)				
<input checked="" type="checkbox"/> The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the Receiving Office) identified above as item(s): <u>1</u>				
<i>* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which the earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.</i>				
Box No. VII INTERNATIONAL SEARCHING AUTHORITY				
Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used): ISA/		Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority): Date (day/month/year) Number Country (or regional Office)		
Box No. VIII CHECK LIST; LANGUAGE OF FILING				
This international application contains the following number of sheets: request : 3 description (excluding sequence listing part) : 13 claims : 4 abstract : 1 drawings : 2 sequence listing part of description : 0 Total number of sheets : 23		This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input type="checkbox"/> separate signed power of attorney 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input checked="" type="checkbox"/> other (specify): <u>Form 23/77</u>		
Figure of the drawings which should accompany the abstract:		Language of filing of the international application: ENGLISH		
Box No. IX SIGNATURE OF APPLICANT OR AGENT				
Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request). For the Applicant Gill Jennings & Every <u>JONES, Helen Marjorie Meredith</u>				
Date: 08 January 1999				

For receiving Office use only	
1. Date of actual receipt of the purported international application:	2. Drawings: <input type="checkbox"/> received: <input type="checkbox"/> not received:
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:	
4. Date of timely receipt of the required corrections under PCT Article 11(2):	
5. International Searching Authority (if two or more are competent): ISA/	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid

For International Bureau use only
Date of receipt of the record copy by the International Bureau:



PCT

FEE CALCULATION SHEET

Annex to the Request

For receiving Office use only

International Application No.

Date stamp of receiving Office

Applicant's or agent's
file reference

HMJ03045WO

Applicant

MOBIL OIL COMPANY LIMITED

CALCULATION OF PRESCRIBED FEES

1. TRANSMITTAL FEE £ 55.00 **T**

2. SEARCH FEE £ 812.00 **S**

International search to be carried out by _____
(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search.)

3. INTERNATIONAL FEE

Basic Fee

The international application contains 23 sheets.

first 30 sheets £ 285.00 **b1**

_____ x _____ = _____ **b2**

remaining sheets additional amount

Add amounts entered at b1 and b2 and enter total at B . . . £ 285.00 **B**

Designation Fee

The international application contains 77 designations.

10 x £ 65 = £ 650.00 **D**

number of designation fees payable (maximum 10) amount of designation fee

Add amounts entered at B and D and enter total at I . . . £ 935.00 **I**

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicant is (or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT (if applicable). £ 22.00 **P**

5. TOTAL FEES PAYABLE £ 1824.00

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

TOTAL

☐ The designation fees are not paid at this time.

MODE OF PAYMENT

☐ authorization to charge
deposit account (see below)

☐ bank draft

☐ coupons

☒ cheque

☐ cash

☐ other (specify):

☐ postal money order

☐ revenue stamps

DEPOSIT ACCOUNT AUTHORISATION (this mode of payment may not be available at all receiving Offices)

The RO/ _____ ☐ is hereby authorized to charge the total fees indicated above to my deposit account.

☐ is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.

☐ is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

Deposit Account Number

Date (day/month/year)

Signature

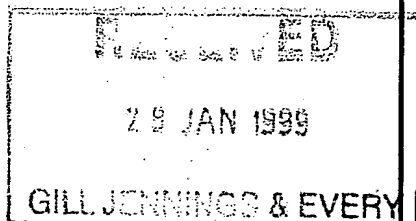
PATENT COOPERATION TREATY

From the RECEIVING OFFICE **Rec'd 07 JUL 2000****PCT**

To:

Gill Jennings & Every
Broadgate House
7 Eldon Street
London

EC2M 7LH

**NOTIFICATION OF THE INTERNATIONAL
APPLICATION NUMBER AND OF THE
INTERNATIONAL FILING DATE**

(PCT Rule 20.5(c))

Date of mailing
(day/month/year)**26.01.99**Applicant's or agents's file reference
HMJ03045WO**IMPORTANT NOTIFICATION**International application No.
PCT/GB99/00064International filing date (day/month/year)
08/01/1999Priority date (day/month/year)
08/01/1998Applicant
Mobil Oil Company Limited et alTitle of the invention
Gypsum Product

1. The applicant is hereby notified that the international application has been accorded the international application number and the international filing date indicated above.

2. The applicant is further notified that the record copy of the international application:

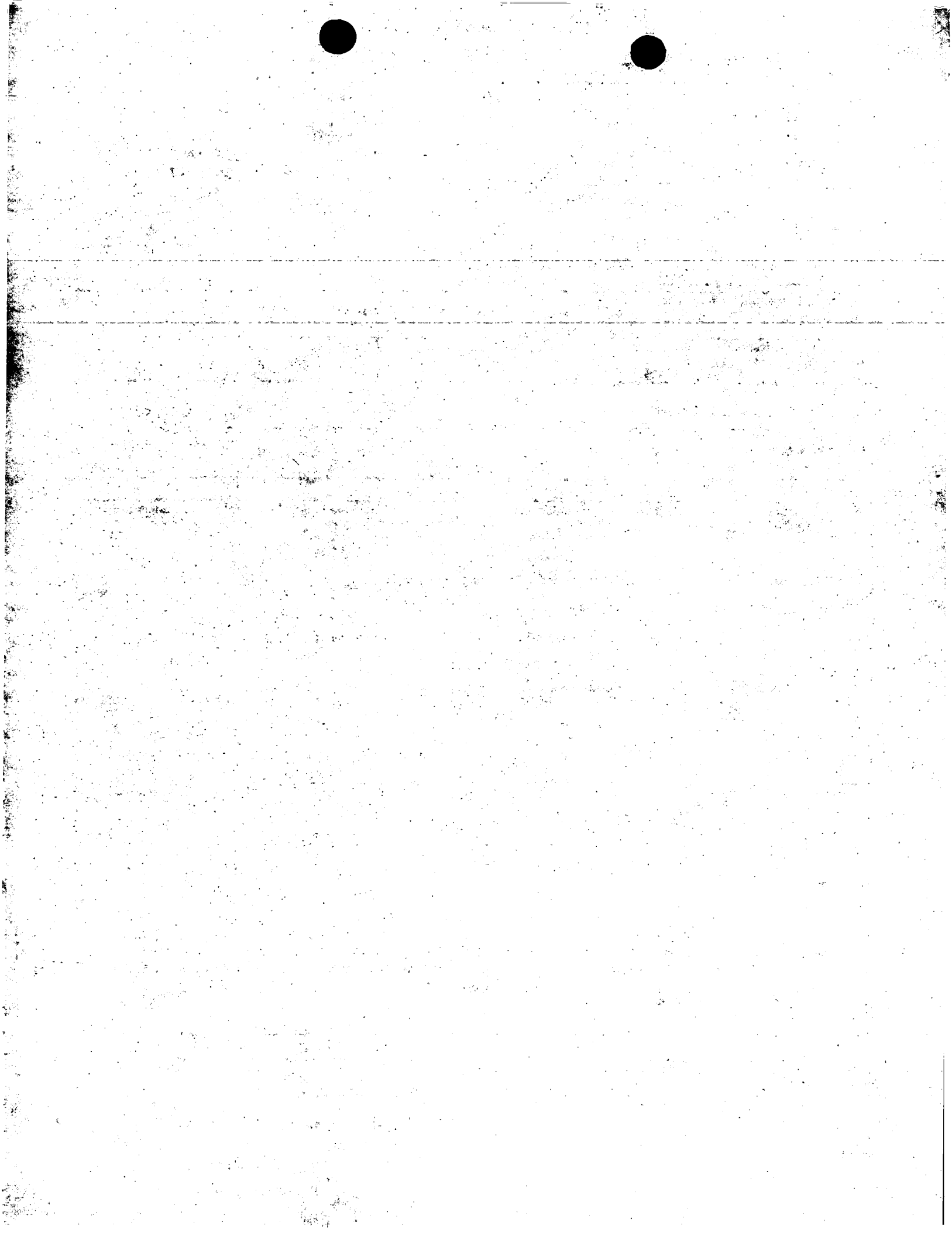
- ☒ was transmitted to the International Bureau on **26.01.99**
- ☐ has not yet been transmitted to the International Bureau for the reason indicated below and a copy of this notification has been sent to the International Bureau*:
- ☐ because the necessary national security clearance has not yet been obtained.
- ☐ because (reason to be specified):

- * The International Bureau monitors the transmittal of the record copy by the receiving Office and will notify the applicant (with Form PCT/IB/301) of its receipt. Should the record copy not have been received by the expiration of 14 months from the priority date, the International Bureau will notify the applicant (Rule 22.1(c)).

Name and mailing address of the receiving Office
The Patent Office
Cardiff Road, Newport
South Wales NP9 1RH
Facsimile No.

Authorized officer

PHIL TREENTelephone No. **01633 814381**



PATENT COOPERATION TREATY

PTO/PCT Rec'd 07 JUL 2000
PCT

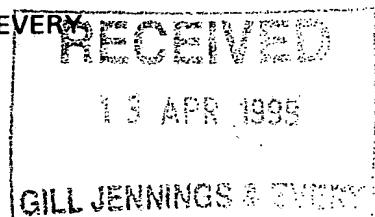
**NOTIFICATION CONCERNING
 SUBMISSION OR TRANSMITTAL
 OF PRIORITY DOCUMENT**

(PCT Administrative Instructions, Section 411)

From the INTERNATIONAL BUREAU

To:

GILL JENNINGS & EVERY
 Broadgate House
 7 Eldon Street
 London EC2M 7LH
 ROYAUME-UNI



Date of mailing (day/month/year) 31 March 1999 (31.03.99)	IMPORTANT NOTIFICATION
Applicant's or agent's file reference HMJ03045WO	
International application No. PCT/GB99/00064	International filing date (day/month/year) 08 January 1999 (08.01.99)
International publication date (day/month/year) Not yet published	Priority date (day/month/year) 08 January 1998 (08.01.98)
Applicant MOBIL OIL COMPANY LIMITED et al	

- The applicant is hereby notified of the date of receipt (except where the letters "NR" appear in the right-hand column) by the International Bureau of the priority document(s) relating to the earlier application(s) indicated below. Unless otherwise indicated by an asterisk appearing next to a date of receipt, or by the letters "NR", in the right-hand column, the priority document concerned was submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b).
- This updates and replaces any previously issued notification concerning submission or transmittal of priority documents.
- An asterisk(*) appearing next to a date of receipt, in the right-hand column, denotes a priority document submitted or transmitted to the International Bureau but not in compliance with Rule 17.1(a) or (b). In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.
- The letters "NR" appearing in the right-hand column denote a priority document which was not received by the International Bureau or which the applicant did not request the receiving Office to prepare and transmit to the International Bureau, as provided by Rule 17.1(a) or (b), respectively. In such a case, the attention of the applicant is directed to Rule 17.1(c) which provides that no designated Office may disregard the priority claim concerned before giving the applicant an opportunity, upon entry into the national phase, to furnish the priority document within a time limit which is reasonable under the circumstances.

<u>Priority date</u>	<u>Priority application No.</u>	<u>Country or regional Office or PCT receiving Office</u>	<u>Date of receipt of priority document</u>
08 Janu 1998 (08.01.98)	9800368.4	GB	29 Marc 1999 (29.03.99)

The International Bureau of WIPO
 34, chemin des Colombettes
 1211 Geneva 20, Switzerland

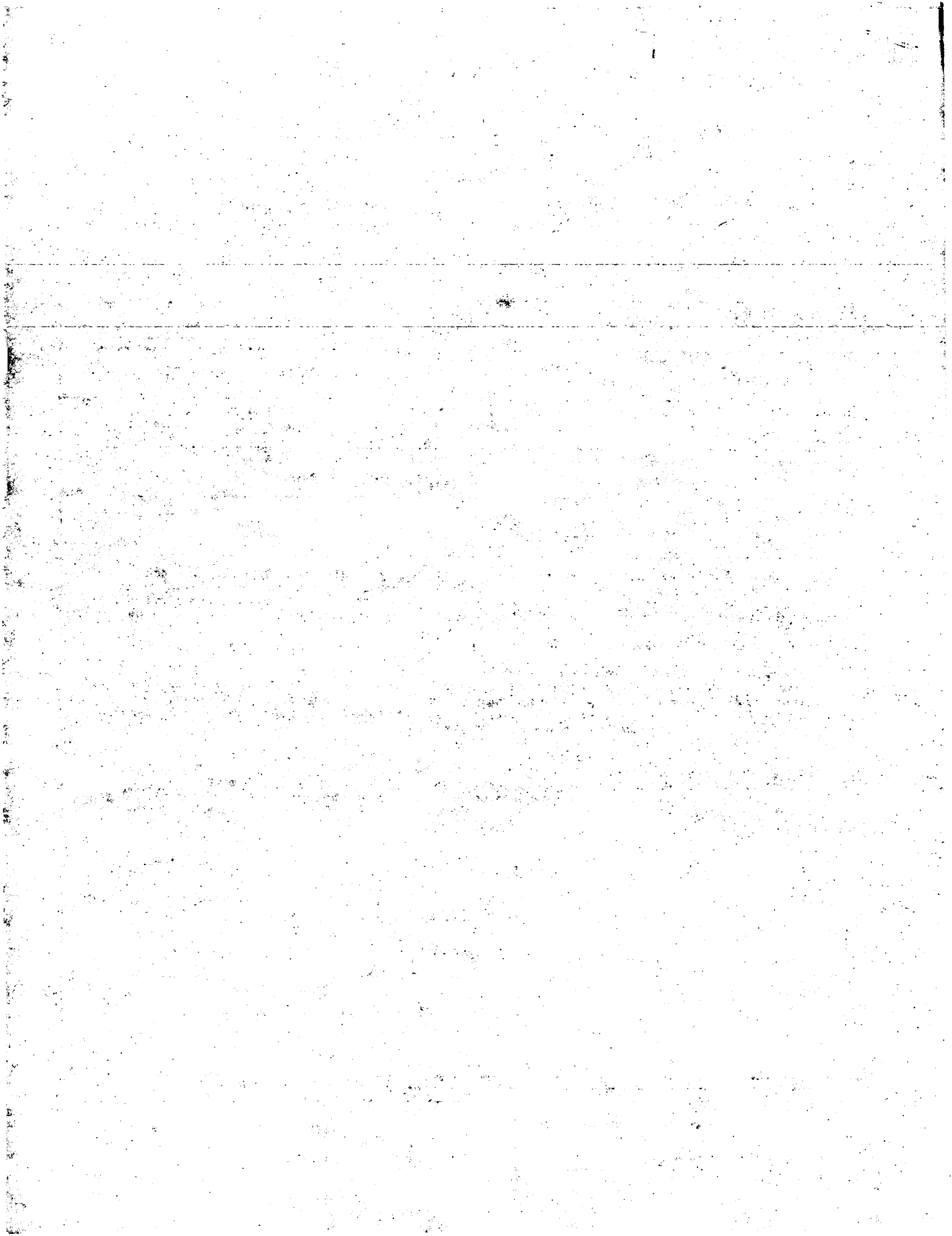
Facsimile No. (41-22) 740.14.35

Authorized officer

Juan Cruz

Telephone No. (41-22) 338.83.38

002548364



PATENT COOPERATION TREATY

PTO/PCT Rec'd 07 JUL 2000
PCT

INFORMATION CONCERNING ELECTED
OFFICES NOTIFIED OF THEIR ELECTION

(PCT Rule 61.3)

From the INTERNATIONAL BUREAU

To:

GILL JENNINGS & EVERY
Broadgate House
7 Eldon Street
London EC2M 7LH
ROYAUME-UNI

Date of mailing (day/month/year) 26 January 2000 (26.01.00)		
Applicant's or agent's file reference HMJ03045WO		IMPORTANT INFORMATION
International application No. PCT/GB99/00064	International filing date (day/month/year) 08 January 1999 (08.01.99)	
Priority date (day/month/year) 08 January 1998 (08.01.98)		
Applicant MOBIL OIL COMPANY LIMITED et al		

1. The applicant is hereby informed that the International Bureau has, according to Article 31(7), notified each of the following Offices of its election:

AP : GH, GM, KE, LS, MW, SD, SZ, UG, ZW

EP : AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE

National : AU, BG, BR, CA, CN, CZ, DE, IL, JP, KP, KR, MN, NO, NZ, PL, RO, RU, SE, SK, US

2. The following Offices have waived the requirement for the notification of their election; the notification will be sent to them by the International Bureau only upon their request:

EA : AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

OA : BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

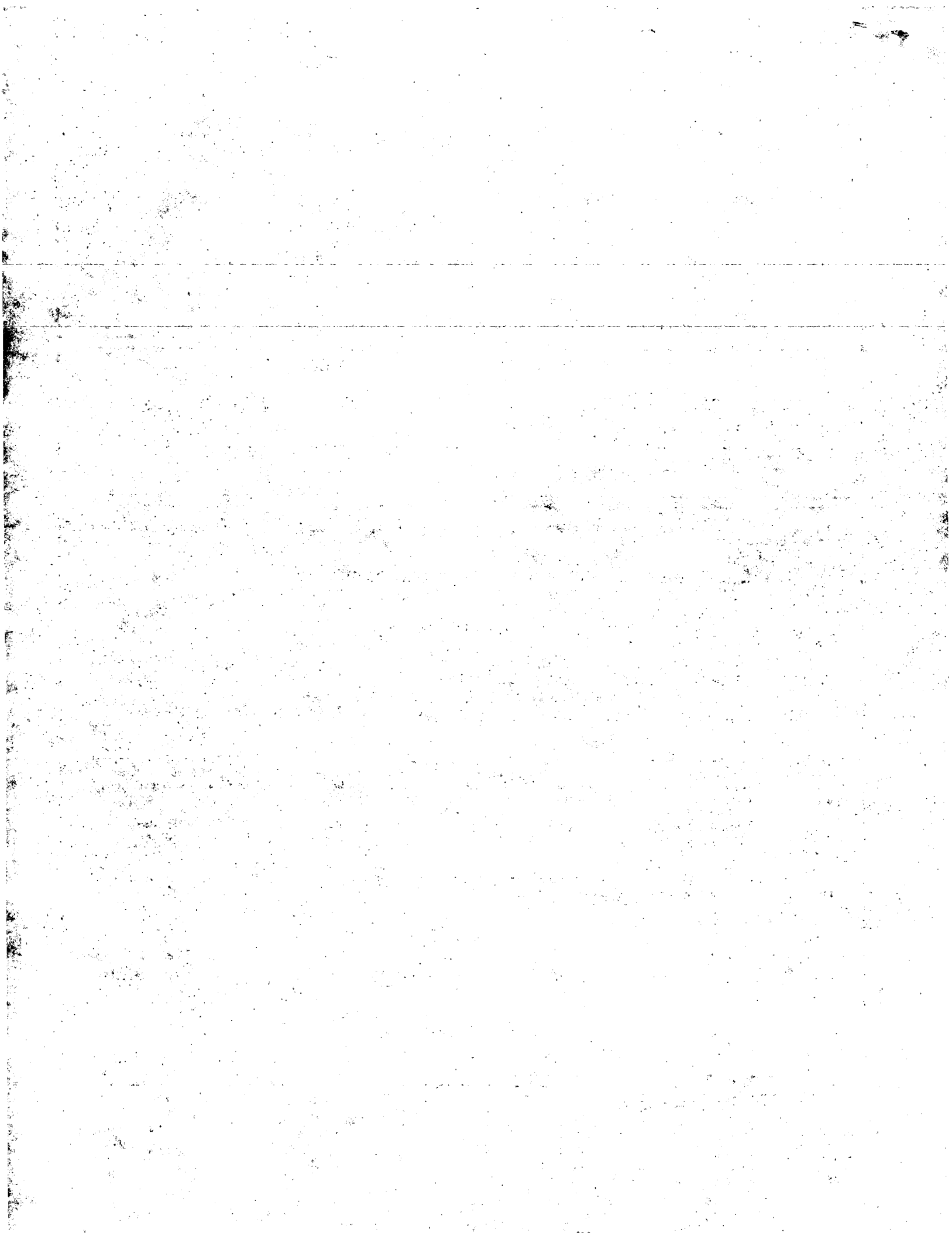
National : AL, AM, AT, AZ, BA, BB, BY, CH, CU, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IN, IS, KE, KG, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MW, MX, PT, SD, SG, SI, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZW

3. The applicant is reminded that he must enter the "national phase" before the expiration of 30 months from the priority date before each of the Offices listed above. This must be done by paying the national fee(s) and furnishing, if prescribed, a translation of the international application (Article 39(1)(a)), as well as, where applicable, by furnishing a translation of any annexes of the international preliminary examination report (Article 36(3)(b) and Rule 74.1).

Some offices have fixed time limits expiring later than the above-mentioned time limit. For detailed information about the applicable time limits and the acts to be performed upon entry into the national phase before a particular Office, see Volume II of the PCT Applicant's Guide.

The entry into the European regional phase is postponed until 31 months from the priority date for all States designated for the purposes of obtaining a European patent.

<p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No. (41-22) 740.14.35</p>	<p>Authorized officer:</p> <p>Olivia RANAIVOJAONA</p> <p>Telephone No. (41-22) 338.83.38</p>
---	--



PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

GILL JENNINGS & EVERY
Broadgate House
7 Eldon Street
London EC2M 7LH
GRANDE BRETAGNE

RECEIVED

12 APR 2000

GILL JENNINGS & EVERY

PCT

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL PRELIMINARY
EXAMINATION REPORT

(PCT Rule 71.1)

Date of mailing
(day/month/year)

10.04.00

Applicant's or agent's file reference
HMJ03045WO

IMPORTANT NOTIFICATION

International application No.
PCT/GB99/00064

International filing date (day/month/year)
08/01/1999

Priority date (day/month/year)
08/01/1998

Applicant

MOBIL OIL COMPANY LIMITED et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/



European Patent Office
D-80298 Munich
Tel. +49 89 2399 - 0 Tx: 523656 epmu d
Fax: +49 89 2399 - 4465

Authorized officer

Myers, J

Tel. +49 89 2399-8111



PATENT COOPERATION TREATY

PCT

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference HMJ03045WO	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB99/00064	International filing date (<i>day/month/year</i>) 08/01/1999	Priority date (<i>day/month/year</i>) 08/01/1998
International Patent Classification (IPC) or national classification and IPC C04B28/14		
Applicant MOBIL OIL COMPANY LIMITED et al.		
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 4 sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of sheets.</p>		
<p>3. This report contains indications relating to the following items:</p> <ul style="list-style-type: none"> I <input checked="" type="checkbox"/> Basis of the report II <input type="checkbox"/> Priority III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability IV <input type="checkbox"/> Lack of unity of invention V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement VI <input type="checkbox"/> Certain documents cited VII <input checked="" type="checkbox"/> Certain defects in the international application VIII <input type="checkbox"/> Certain observations on the international application 		
Date of submission of the demand 02/06/1999	Date of completion of this report <div style="text-align: center; font-size: 1.2em;">10.04.00</div>	
Name and mailing address of the international preliminary examining authority: <div style="display: flex; align-items: center;"> <div> European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465 </div> </div>	Authorized officer Harbron, J Telephone No. +49 89 2399 8453	



**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB99/00064

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*):

Description, pages:

1-13 as originally filed

Claims, No.:

1-20 as originally filed

Drawings, sheets:

1/2-2/2 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)).

4. Additional observations, if necessary:

INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/GB99/00064

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	1-20
	No:	Claims	
Inventive step (IS)	Yes:	Claims	1-20
	No:	Claims	
Industrial applicability (IA)	Yes:	Claims	1-20
	No:	Claims	

2. Citations and explanations

see separate sheet

VII. Certain defects in the international application

The following defects in the form or contents of the international application have been noted:

see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB99/00064

Re Item V

Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Reference is made to the following documents:

D1: US-A-5695553

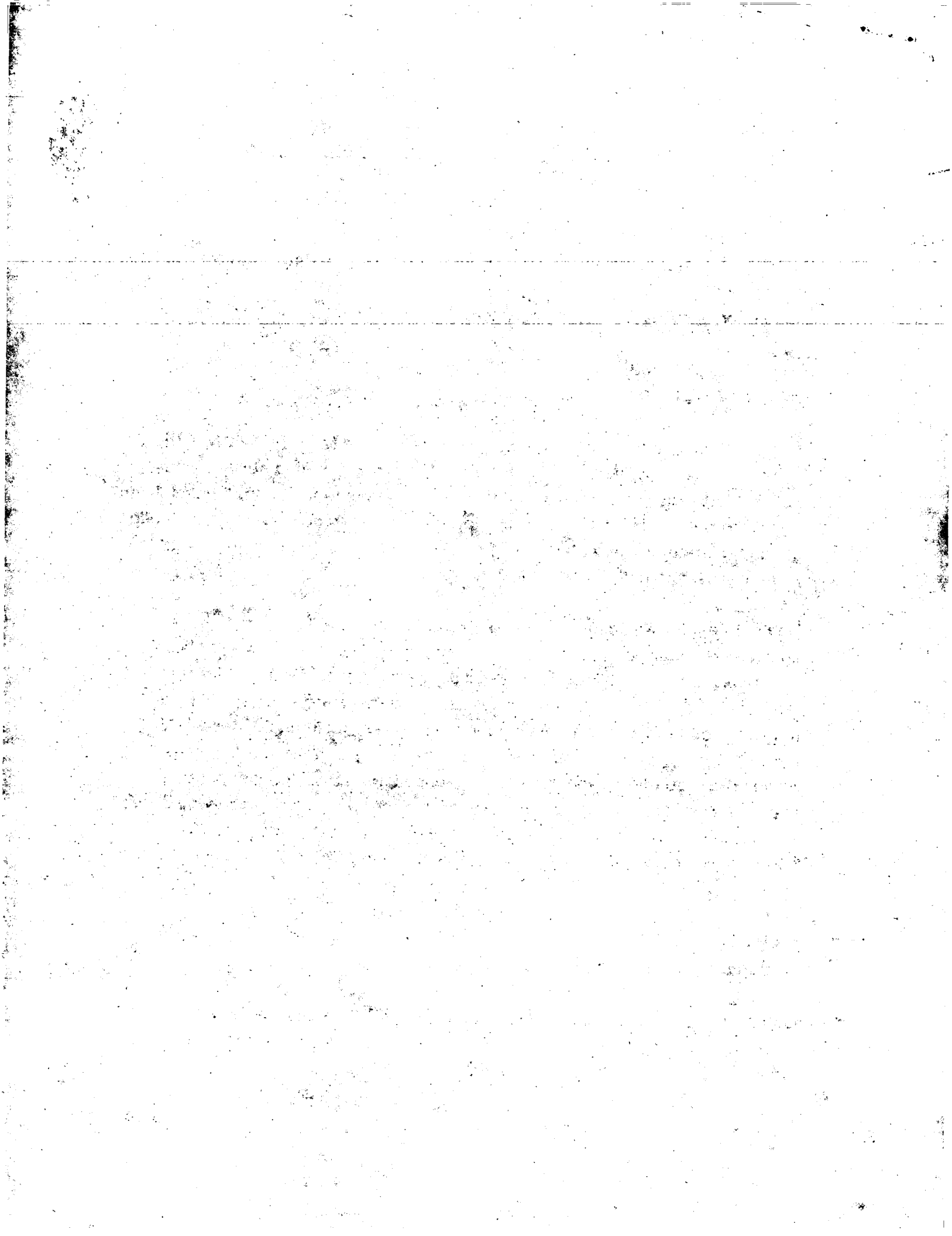
D2: US-A-4315957

2. None of the cited prior art documents discloses all the features of any of the independent claims. In particular, the cloud point and foaming ability of the nonionic surfactant are not given in the prior art documents, so that these claims appear to be novel (Art. 33(2) PCT). However, it should be noted that the absence of certain properties of materials in a disclosure does not necessarily mean that said materials do not possess such characteristics.
3. Both of the prior art documents cited above disclose emulsifier systems which comprise nonionic surfactants and sulphated anionic dispersants. D1 discloses a poly(phenolate carboxylate) resin emulsifier with a lignosulfonic acid dispersant (column 3, lines 40-59) and D2 also teaches the use of nonionic emulsifiers which may be combined with sulphated anionic emulsifiers (column 3, lines 34-45). Neither of the prior art documents disclose nor suggest an emulsifier system having the properties given in the characterising portion of the independent claims. Furthermore, there is no reference in either document which would lead the skilled man to combine the teachings of one with that of the other. The application is considered to involve an inventive step (Art. 33(3) PCT).

Re Item VII

Certain defects in the international application

1. Contrary to the requirements of Rule 5.1(a)(ii) PCT, the relevant background art disclosed in the documents D1 and D2 is not mentioned in the description, nor are these documents identified therein.



PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C04B 28/14, C08L 91/06 // (C04B 28/14, 24:08, 24:22, 24:32, 24:36), 111:27		(11) International Publication Number: WO 99/35103		
A1		(43) International Publication Date: 15 July 1999 (15.07.99)		
<table border="1"><tr><td><p>(21) International Application Number: PCT/GB99/00064</p><p>(22) International Filing Date: 8 January 1999 (08.01.99)</p><p>(30) Priority Data: 9800368.4 8 January 1998 (08.01.98) GB</p><p>(71) Applicant (for all designated States except US): MOBIL OIL COMPANY LIMITED [GB/GB]; Mobil House, 500-600 Witan Gate, Central Milton Keynes, Buckinghamshire MK9 1ES (GB).</p><p>(72) Inventor; and</p><p>(75) Inventor/Applicant (for US only): BOELEEE, Scotia [NZ/GB]; 105 Forest Road, Tunbridge Wells, Kent TN2 5BG (GB).</p><p>(74) Agent: GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).</p></td><td><p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p><p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p></td></tr></table>			<p>(21) International Application Number: PCT/GB99/00064</p> <p>(22) International Filing Date: 8 January 1999 (08.01.99)</p> <p>(30) Priority Data: 9800368.4 8 January 1998 (08.01.98) GB</p> <p>(71) Applicant (for all designated States except US): MOBIL OIL COMPANY LIMITED [GB/GB]; Mobil House, 500-600 Witan Gate, Central Milton Keynes, Buckinghamshire MK9 1ES (GB).</p> <p>(72) Inventor; and</p> <p>(75) Inventor/Applicant (for US only): BOELEEE, Scotia [NZ/GB]; 105 Forest Road, Tunbridge Wells, Kent TN2 5BG (GB).</p> <p>(74) Agent: GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).</p>	<p>(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).</p> <p>Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>
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(54) Title: GYPSUM PRODUCT				
(57) Abstract				
<p>A wax emulsion comprising an emulsifying system containing a sulphated anionic surfactant and a non ionic surfactant having high water solubility (cloud point) and high foaming ability is added to a gypsum slurry to improve the moisture resistance of gypsum board. The wax is a mixture of a petroleum derived hydrocarbon wax and a montan wax.</p>				

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GYPSUM PRODUCT

This invention relates to a gypsum product and to a process for its manufacture. More particularly, this invention relates to a foamed gypsum product of improved water resistance and/or reduced density and to a process, preferably to a continuous process, for its manufacture.

Gypsum board (or plaster board or wallboard) is used extensively in the construction industry. It typically comprises a substantially flat core of set gypsum on either side of which a liner may be adhered. A liner typically comprises paper. The core may be reinforced; for example, reinforced with glass fibres.

Gypsum products (or Plaster of Paris or plaster products) are produced by mixing anhydrous calcium sulphate or calcium sulphate hemihydrate with water, and permitting the mixture to set thereby producing calcium sulphate dihydrate. Often the slurry is foamed by incorporating a preformed solution of foaming agent in water (a surface active material) before adding to the mould means. A pervasive problem with gypsum products, however, is that calcium sulphate dihydrate absorbs water and this reduces the strength of the gypsum product. Because of this, plaster board (for example) is required, at least in uses where a relatively high humidity is anticipated (for example, kitchens or bathrooms) to be substantially moisture resistant and this requires the presence of a hydrophobing agent. ("Hydrophobing" is a term used in the art to denote a method of preventing, or reducing water absorption).

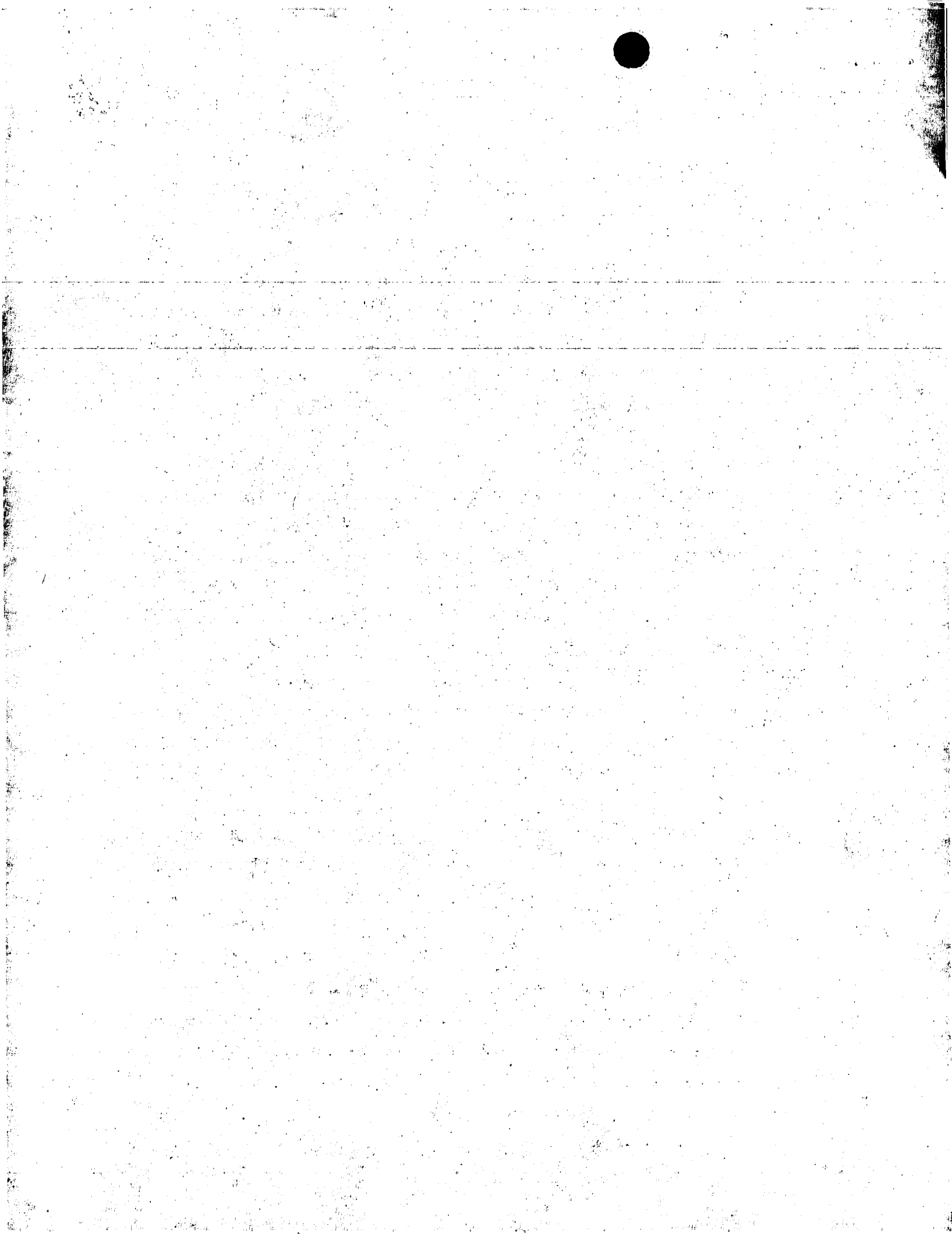
Silicone oil has previously been used as a hydrophobing agent for gypsum products. It is, however, expensive and in relatively short supply. It also has

difficulty in providing a moisture resistance of less than 5 wt % water absorption in the test hereinafter mentioned.

In US-A-5437722 an aqueous emulsion comprising a hydrocarbon wax, a montan wax and emulsifier/stabiliser system and also including a polyvinyl alcohol, is used to render gypsum products water resistant. The emulsifier system may include non-ionic or anionic surfactant and alkali. Examples of non ionic surfactants are alkyl-phenoxypoly(ethyleneoxy) ethanols, sorbitan fatty acid esters and polyoxyethylene sorbitan fatty acid esters. Examples of anionic surfactants are saponified fatty acids.

In our copending application number PCT/GB97/02366, unpublished at the priority date of the present invention, we describe a foamed gypsum product which is hydrophobed by incorporation of an aqueous emulsion comprising a hydrocarbon wax, a montan wax and a colloid stabilised emulsifier system. The preferred colloid stabilised emulsifier system comprises either organic or, more preferably, inorganic colloidal materials. One example is, a montmorillonite clay based system in combination with a sodium naphthalene sulphonate.

In the above mentioned PCT application, a comparative example uses an emulsifier system based on a combination of a nonionic surfactant, which was not specifically defined, with an anionic surfactant again, not specifically defined. The emulsifier system used in that comparative example was in fact a combination of an alkyl phenyl ethoxylate with a soap-type anionic surfactant. The worked example using that emulsifier system did not work. In fact it is now believed that the anionic surfactant caused collapse of the foam or that, upon addition to a slurry formed using



relatively hard water, the anionic surfactant was precipitated out of the system by the hardness ions.

The above mentioned PCT application also discloses a comparative example using a nonionic surfactant based emulsifier system including no anionic emulsifier. Whilst this gave some improvement over the mixed system, it was found that using a high enough level of emulsion in the gypsum product to achieve adequate density and/or water adsorption figures lead to over-wetting of paper used for the gypsum product and delamination during production.

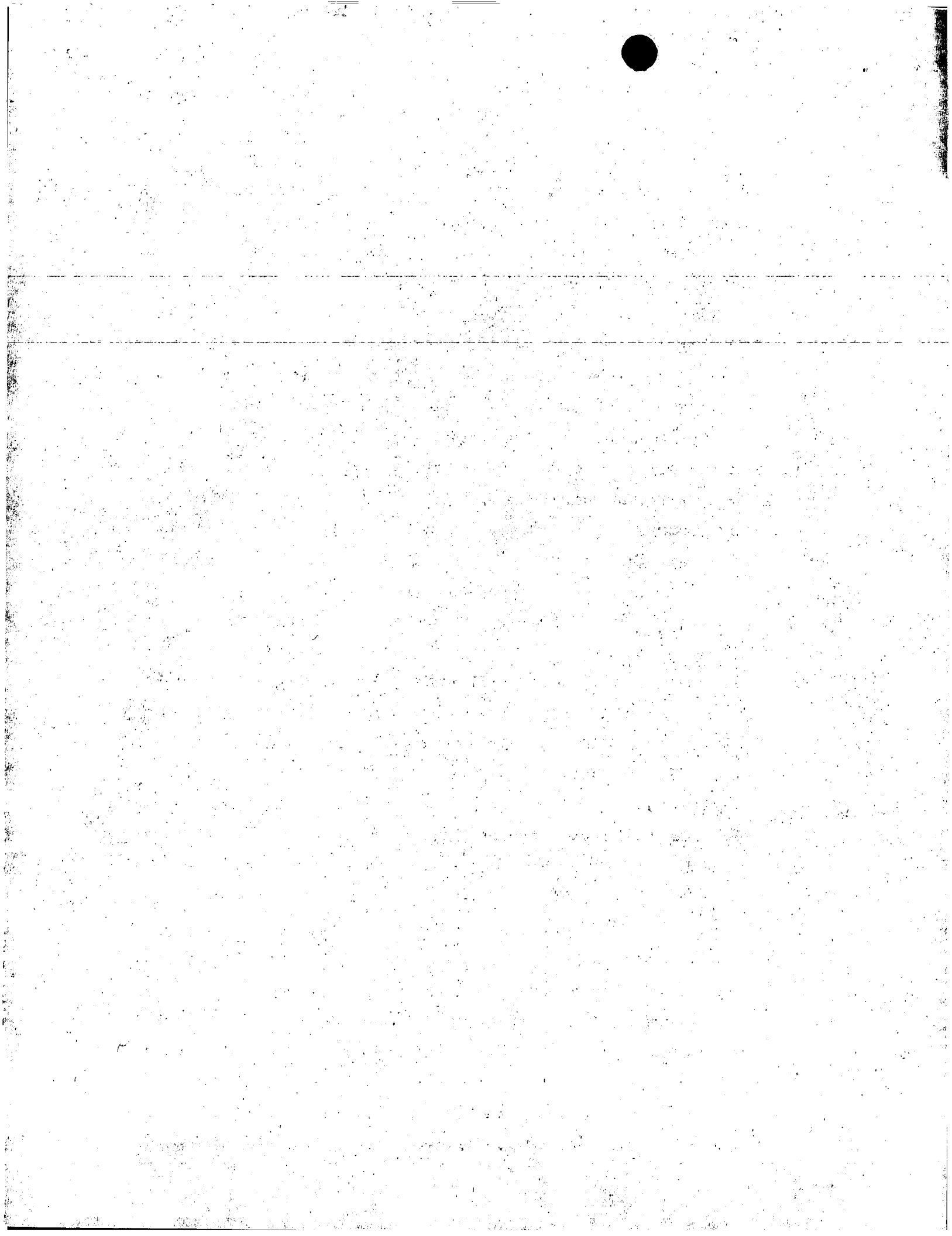
In the present invention there is provided a process for producing a foamed gypsum product involving the following steps:

- a) a slurry of gypsum is formed in water
- b) the slurry is introduced to mould means and allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point (in saline per DIN 53917) of at least 50; and
- ii) an anionic dispersing agent which is a sulphated compound.

Preferably the anionic dispersant is a so called acid stable compound, that is the compound is ionised over a wide range of pH's including acidic pH. The acid stability can be judged by the pK_a of the conjugate acid, which should preferably be less than 4, more preferably less than



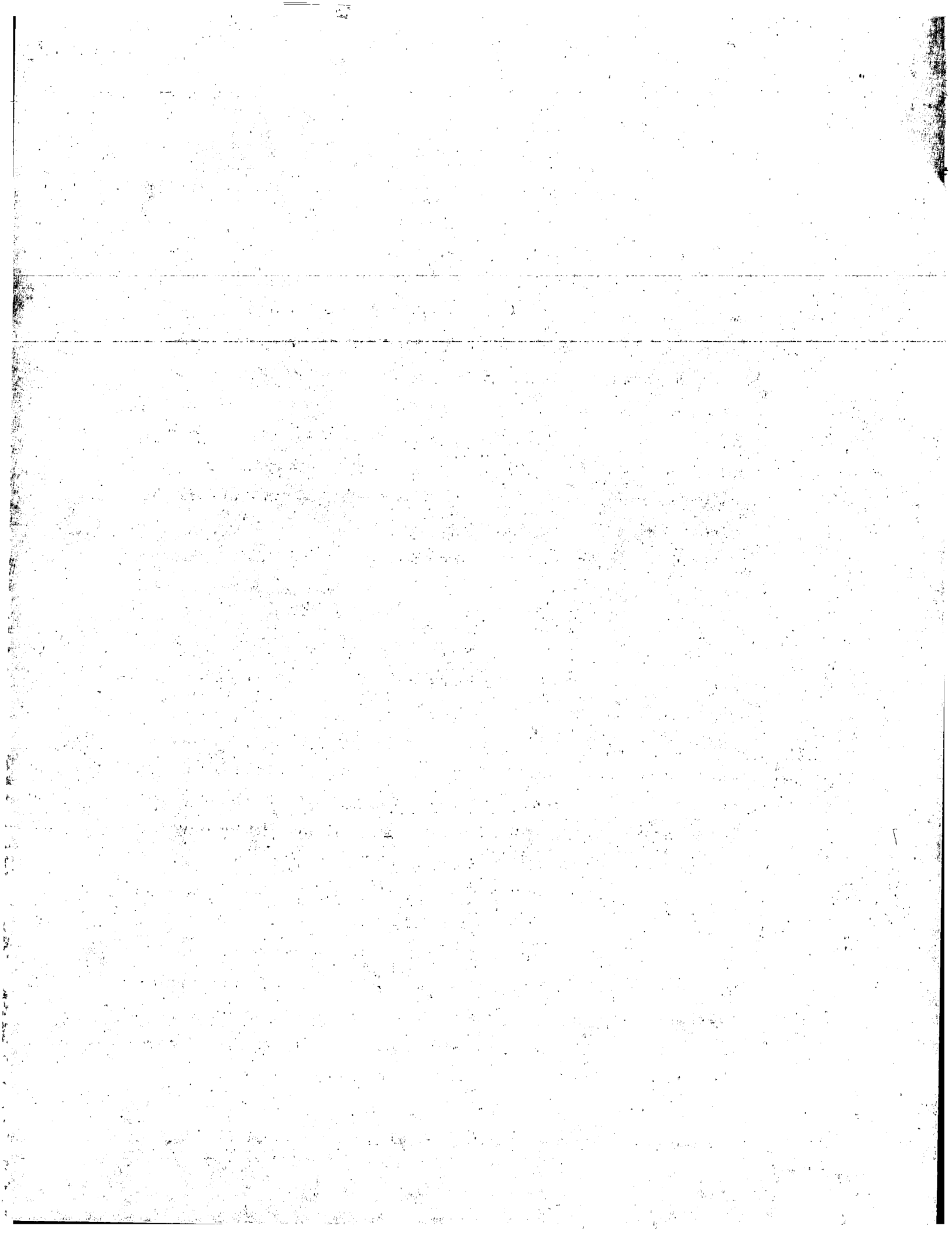
3, for instance 2 or less. The anionic dispersant is generally a sulphate or a sulphonate.

A suitable class of anionic dispersants are sulphated naphthalene/formaldehyde condensates, for instance having
5 molecular weight in the range 6000 to 40000. These compounds are also known as naphthalene sulphonates. Other aryl sulphonates may also be used. The anionic dispersant is generally used in the emulsion in the form of its sodium salt. Alternatively potassium, ammonium, or even divalent
10 metal salts such as calcium or magnesium, may be used. Suitable compounds are available from BASF AG under the trade name Tamol (trademark).

The nonionic surfactant must be relatively water soluble. The water solubility of non ionic surfactants can
15 be determined by standard test method DIN 53917 in saline. The component should have a cloud point of at least 50, for instance more than 60, up to around 100, for instance approximately 75.

We have found that the nonionic surfactant giving
20 optimum performance is one which has a high foaming ability. Foaming ability can be measured by standard test methods DIN 53902. For instance the test should be carried out according to the method given in sheet 1 of that standard test method, at 40°C, with the surfactant being
25 used in a concentration of 2 g/l in water containing 1.8 mmol Ca ions/l, the duration of the test being 30 seconds. The foaming ability should be at least 300, more preferably at least 500, for instance up to 750. Nonionic surfactants with foaming ability using the above mentioned test method
30 of around 600 are available.

The nonionic surfactant is generally an ethoxylated higher alkyl, alkenyl, alkanoyl or alkenoyl compound.



Alternatively ethoxylated aryl compounds may be used, for instance ethoxylated alkyl phenol derivatives. Preferably the compound is a C_{8-18} -alkanol ethoxylated with 3 to 30 equivalents of ethylene oxide, for instance a C_{12-18} -alkanol ethoxylated with 10 to 20 equivalents of ethylene oxide. Suitable compounds are available under the trade name Lutensol (trademark) from BASF AG. It may alternatively be possible to use polyglycosylated alkyl, alkenyl, alkanoyl, alkenoyl and aryl compounds for instance alkyl polyglucosides.

The use of an acid stable anionic dispersant, it is believed, avoids the addition of the emulsion resulting in the collapse of the foam in the gypsum slurry. Accordingly the density of the product is optimised. The use of the anionic dispersant in combination with non ionic surfactant avoids the use of such high concentrations of nonionic dispersant in the gypsum slurry which can lead to over-wetting of paper used in the gypsum product and delamination during production. The preferred surfactant makes the emulsion suitable for use with gypsum slurries made up in hard water, for instance water having hardness value of at least 100 ppm Ca^{2+} even more than 150 ppm Ca^{2+} , for instance at least 200 ppm Ca^{2+} .

The use of the high foaming non ionic surfactant is believed also to contribute to optimisation of the gypsum slurry foam and the density of the final product. The preferred combination of surfactants in the emulsifier system allows high levels of wax to be incorporated into the final product for optimum hydrophobing of the gypsum product.

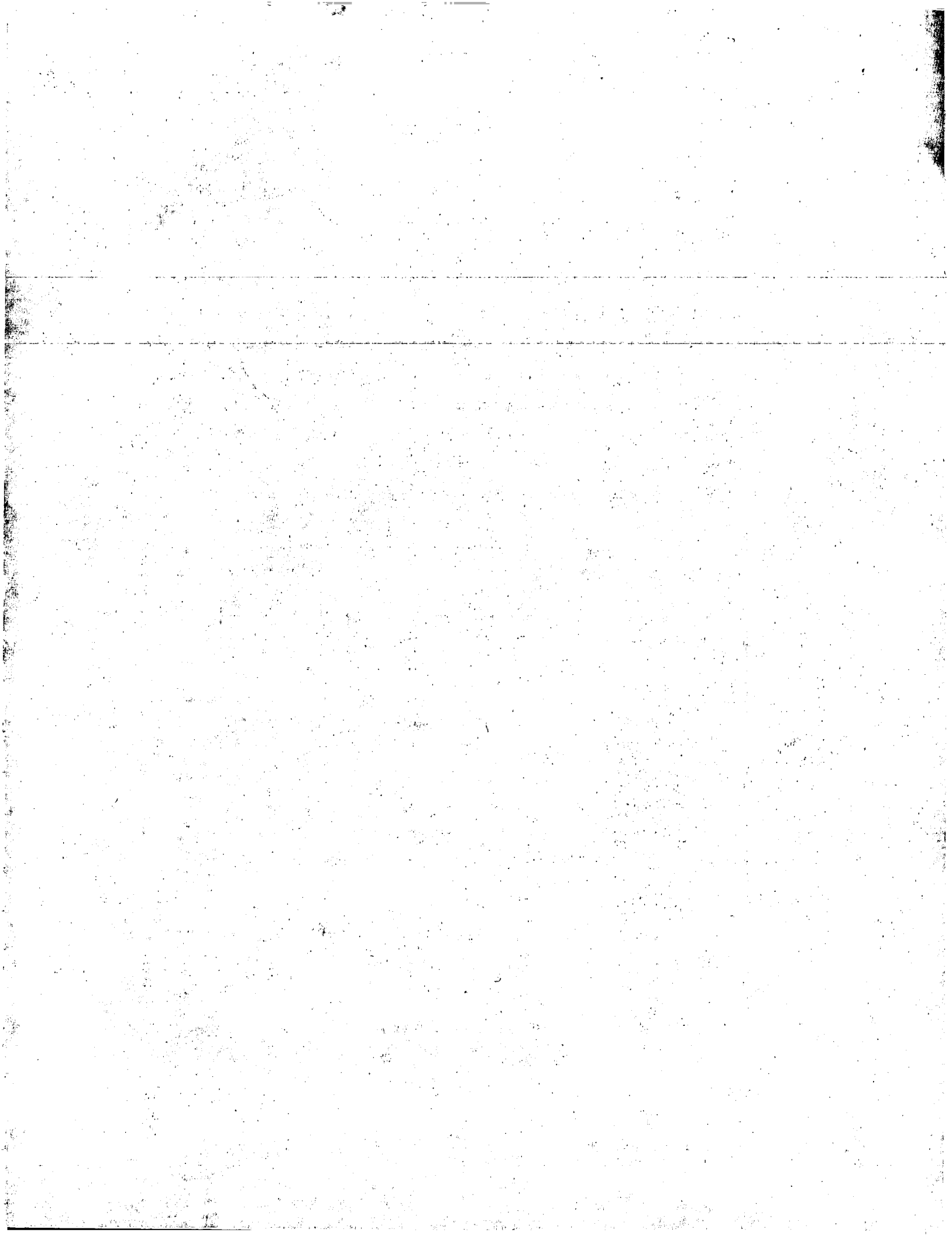
The emulsifier system has process advantages also in production of non-foamed products, for instance, other

products made on continuous lines such as fire resistance board. The system provides good compatibility with the equipment

The petroleum-derived hydrocarbon wax (a) is preferably one with a high melting point and a low oil content. A preferred such wax is a paraffin wax, such as fully refined paraffin wax. Fully refined paraffin waxes are generally obtained from highly paraffinic refinery streams such as those obtained from the solvent dewaxing of distillates and other lube fractions. The product is further typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION	
		MIN	MAX
Congealing Point (°C)	ASTM D938	55	69
Oil in Wax (%)	ASTM D721		1
Penetration at 25°C (mm/10)	ASTM D1321	10	20
Penetration at 50°C (mm/10)	ASTM D1321		80
Viscosity (cSt @ 100°C)	ASTM D445	3	7

An example of a fully refined paraffin wax which has been found to be entirely satisfactory, and which satisfies the above specification, is MOBILWAX 135 (derived from the 150 SPN stream) as supplied by Mobil Oil Company Limited; MOBILWAX 145 or 150 (derived from the 300 or 450 SPN stream) are also suitable. While these waxes are hydrofinished to give a white colour and good odour, unfinished wax (which differs only in colour and odour) is also suitable for use in accordance with the invention.



The petroleum-derived hydrocarbon wax a) suitably comprises from 20 to 40 wt % of the aqueous emulsion, preferably from 25 to 35 wt % of the aqueous emulsion.

The montan wax or lignite wax b) is another wax with a high melting point. It is preferably used in crude (or raw) form. Such a product is typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION		PREFERRED SPECIFICATION	
		MIN	MAX	MIN	MAX
Congealing Point (°C)	ASTM D938	67	80	75	80
Acid Value (mg KOH/g)	ASTM D1980	10	37	10	20
Saponification Value (mgKOH/g)	ASTM D1962	35	100	65	90
Ash Content (% wt)	ASTM D482		1		1.0
Density at 20°C (g/cm³)	ASTM D1298	0.95	1.04	0.95	1.04
Viscosity (cSt at 90°C)	ASTM D445	20	400	150	400
Viscosity (cSt at 100°C)	ASTM D445	20	200	60	150

The montan wax b) suitably comprises from 10 to 20 wt % of the aqueous emulsion, preferably from 11 to 15 wt % of the aqueous emulsion.

The emulsifier system (i.e. the total of two or more components of a multi-component system) is suitably present in an amount from 0.5 to 6 wt %, preferably 1.2 to 5 wt %, more preferably 1.5 to 4% of the aqueous emulsion. The

ratio of the non-ionic and anionic components is preferably in the range 5:1 to 1:5, more preferably 3:1 to 1:3, most preferably 2:1 to 1:2.

In the invention the slurry in water, preferably
5 contains 100 parts by weight of gypsum and from 0.5 to 10, preferably from 1 to 5 % by weight of an emulsion as herein defined. The slurry suitably contains 50-60 weight % gypsum and 40-50 weight % water, preferably about 55% gypsum. An accelerator is usually added, for instance a
10 slurry mix from a previous batch.

The slurry preferably contains a foaming agent. Preferably the product is a paper lined board and the process thus preferably includes a step of foaming, usually involving formation of a pre-formed foam by vigorous
15 stirring of the foaming agent in water, followed by mixing the prefoam into preformed gypsum slurry containing the emulsion.

This invention also provides a water-resistant gypsum product which comprises the set composition. Whilst the
20 product may be an unlined board, the invention is particularly applicable to a product which comprises a core product of a set such composition sandwiched between a pair of liners usually paper liners. Another suitable product is fire resistant board which has a glass fibre scrim
25 embedded in each surface of the gypsum board, which is generally unfoamed. The invention includes also the emulsion itself and the process for making it.

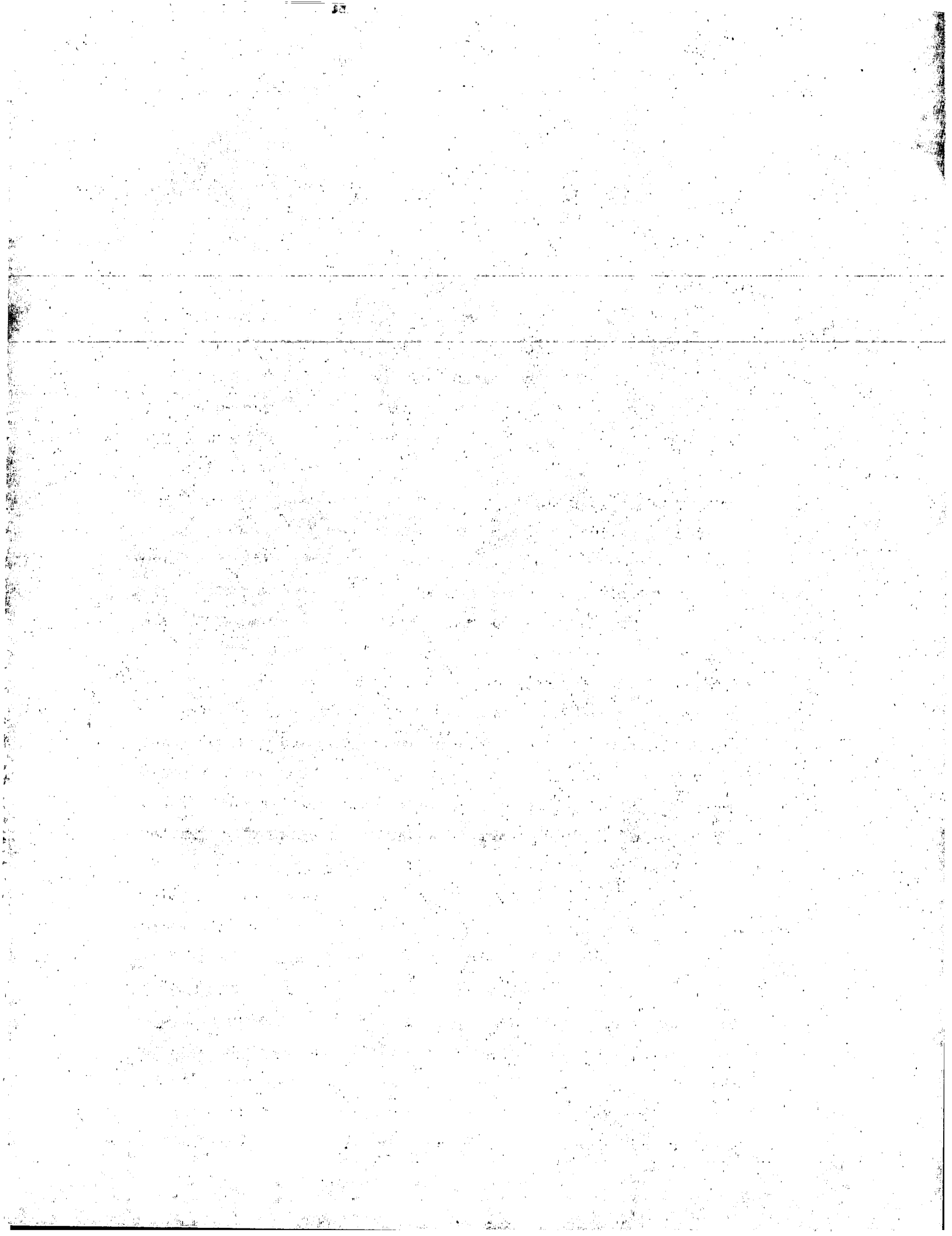
The invention further provides a process for the preparation of a water-resistant gypsum board product,
30 which process comprises forming a mixture which is a slurry in water containing 100 parts by weight of gypsum and from 0.5 to 5 parts by weight of an emulsion according to the

invention; forming a layer of the mixture in a mould means and drying the layer of gypsum mixture, while permitting hydration of the gypsum, for form a board product. Preferably the process is continuous. The process may be
5 for forming tiles or blocks or boards. Blocks may be formed in moulds from which they are removed when set. Tiles or boards may be formed by spreading a layer of the gypsum mixture on a first planar substrate, a second planar substrate is positioned over the layer to form an assembly,
10 and the mixture is allowed to set in the assembly. A gasket may be provided between the planar substrates. Where the product is a lined board, the first and second planar substrates are constituted by liner, for instance paper, usually supported in a mould. Where the product is
15 to be unlined, the planar substrates are removed when the product is set. Where the product is a block, it is usually removed from a mould before the mixture is completely set, but when it is hard enough to handle. Where the product is a fire resistant board a fibreglass
20 scrim is embedded in each side of the slurry in the mould means by feeding to each side of the poured (unfoamed) slurry before the nip forming a dam in an apparatus similar to that shown in Figures 3 and 4 below.

This invention includes the use of an aqueous emulsion
25 of the invention to furnish a gypsum product with water resistance and the use of an aqueous emulsion of the invention to aid foaming of a gypsum slurry, for instance to reduce the density of the set foamed gypsum product.

Figure 1 shows the water absorption results of the
30 product of the comparative example.

Figure 2 shows the water absorption results of the product of the example of the invention.



Figures 3 and 4 are a schematic representations of a foaming station for lined gypsum board.

The following Example illustrate the invention.

EXAMPLE

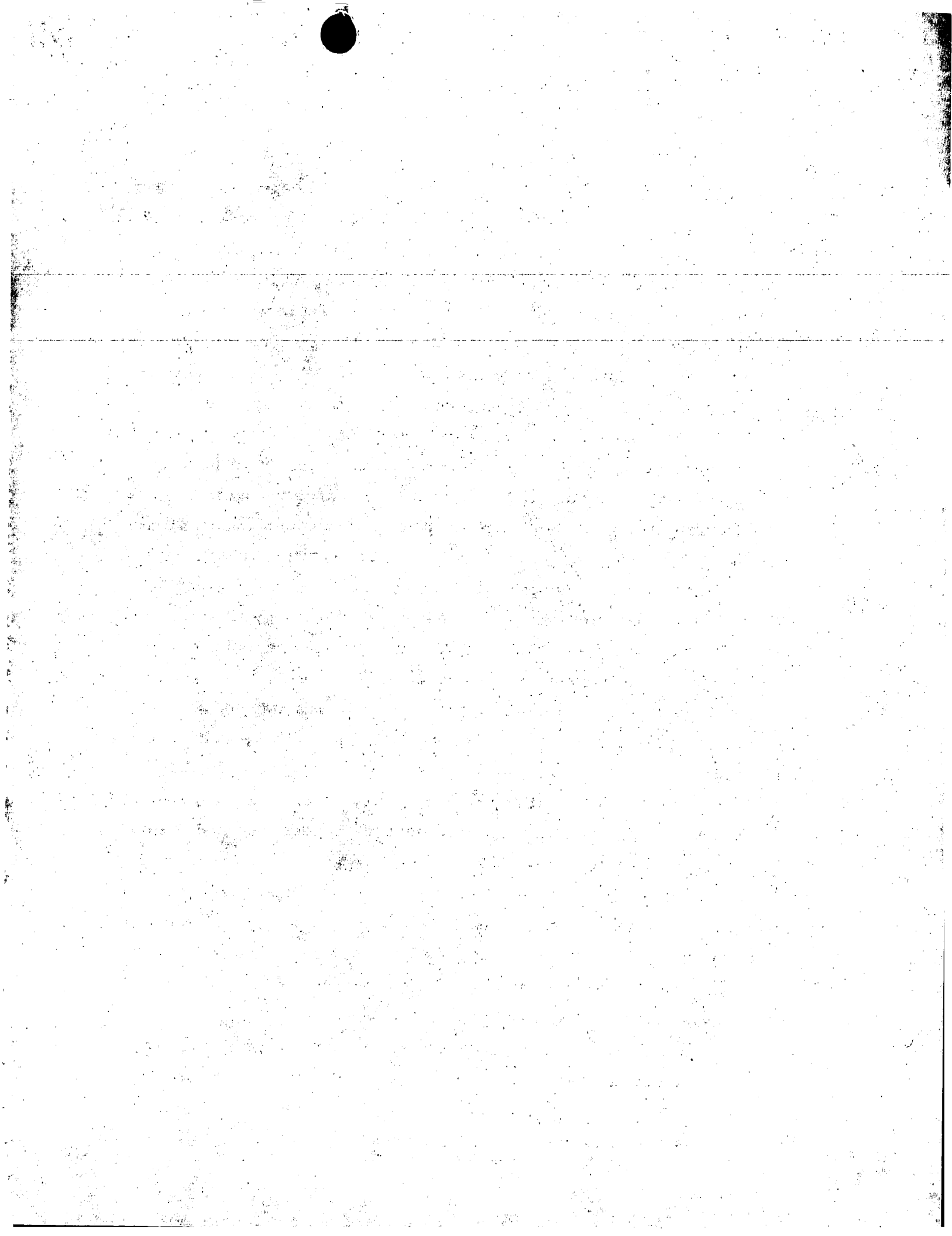
5 First the wax phase was prepared by dissolving 12 wt % crude montan wax (Crude Montan Wax supplied by Schuemann Sabol GmbH) in 30 wt % of fully refined paraffin wax (MOBILWAX 135 supplied by Mobil Oil Company Limited) at a suitable raised temperature. 1% by weight sodium
10 naphthalene sulphonate (a sulphated naphthalene formaldehyde condensate having a molecular weight of about 6000 to 40000) from the TAMOL (trademark) range supplied by BASF was added to water to form the aqueous phase and stirred for a period. 0.45% Non ionic surfactant (added as
15 a 90% aqueous solution) (a C₁₃ alkanol - 12 mole ethoxylate available as Lutensol T012 series) was then added to the aqueous phase and stirred for a period. The temperature of the wax melt was lowered to 100°C and the wax phase was next added to the aqueous phase heated to a suitable
20 temperature, with stirring for a suitable period to form a pre-emulsion. The pre-emulsion still at a raised temperature was next recycled through a homogeniser, with no impressed pressure, for a full pass. Gradually, the pressure was increased to a value in the range 20-25 MPa
25 (220 bar) and the emulsion recycled for a further pass to form an aqueous emulsion in accordance with the invention.

The emulsion was then tested for its performance in the production of a gypsum product. A conventional foaming agent was mixed with vigorous stirring with a suitable
30 quantity water to generate a foam mixture. A gypsum slurry mix was prepared by adding a predetermined amount (1.4, 1.6 or 1.8% by weight based on the amount of gypsum) of wax

emulsion (according to the invention or comparative) to around 40 parts by weight water along with predetermined amounts of a wetting agent, starch and an accelerator in a total amount of 0.38 parts by weight. To this around 58
5 parts by weight gypsum was added with stirring. The pre-generated foam mix was next added to the gypsum slurry and stirred to form a foamed gypsum slurry. The slurry was poured into a paper lined mould of 300 x 300 x 12.5 mm dimension and a second sheet of paper placed on top to form
10 a gypsum coupon which was then dried in three stages of successively lower temperatures and longer times to a constant weight. The density and 2 hour water absorption were then determined. The density was calculated by dividing the dry weight of the test specimen by the mould
15 volume. The water absorption was determined by cutting a test specimen measuring 280 x 280 mm from the coupon and immersing this specimen in a water bath at 23°C covered with 25 to 35 mm of water for 2 hours. Its weight before and after immersion was measured and the percentage
20 increase calculated.

The results, which include comparative tests, are shown in figures 1 and 2. In these tests density and 2 hr water absorption were measured and reported using a the emulsifier system of the invention and also as comparison,
25 based on the colloid emulsified system of the above mentioned PCT publication, in which the same amounts of wax emulsion containing an emulsifier system of a bentonite clay and sodium naphthalene sulphonate.

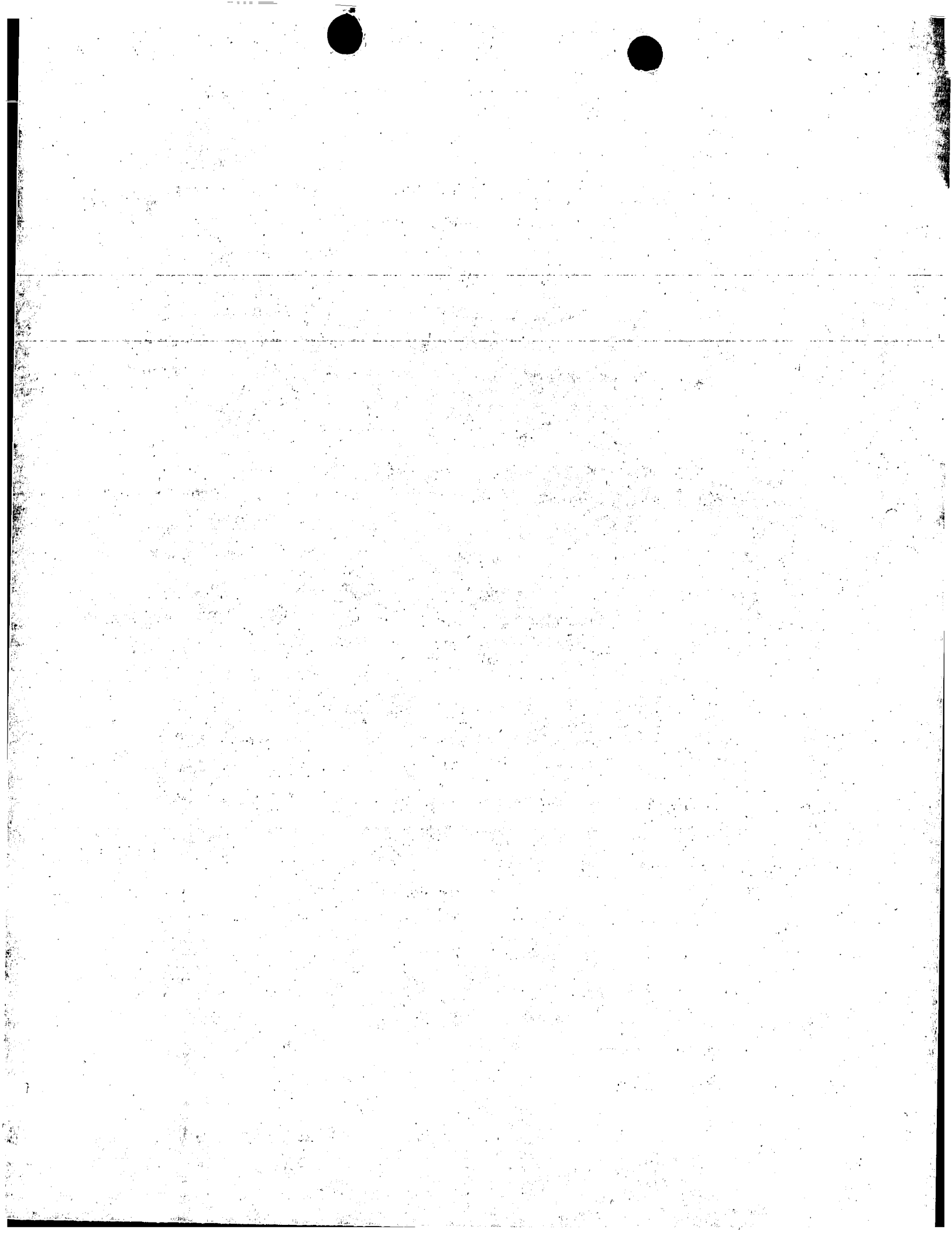
A further example has also been conducted in which the
30 emulsion containing Mobilwax 135, which has a melting point (congealing point) in the range 57-60°C and a maximum content of oil of 1.0wt%, is used at a level of 4% by



weight. This example is then repeated, but using emulsions (at 4% by weight in the gypsum) in which the Mobil wax 135 is replaced by waxes having higher (63-66°C , and 66-69°C, respectively) and lower (54-57°C) melting/congealing points. When used at the same levels in the emulsion, the emulsion (at the same level in the gypsum) produced good results for water absorption. The values were less than 5%, indeed less than 2.5%, in each case.

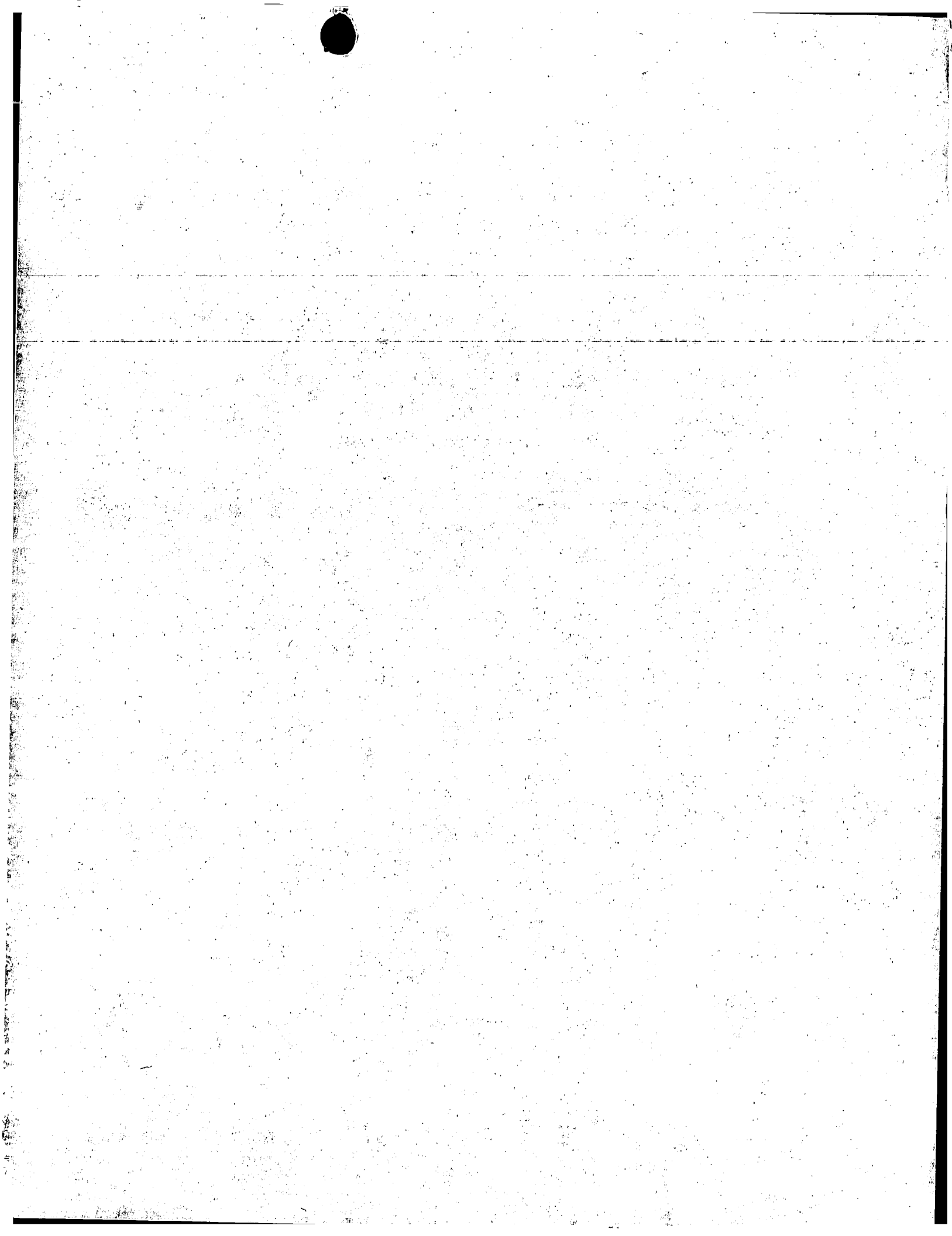
In Figures 3 and 4 there is shown a forming station for gypsum board manufacture is shown generally at 100. It comprises a conveyor 1 which is formed from an array of like, generally coplanar, parallel driven rollers 2 which are rotatable in the same sense. Above the conveyor is a manifold mixing box 3 into which entry conduits 4 and 5 and a plurality of exit nozzles 6,6',6" are flowably connected. A contra-rotatable roller 7 is biased toward rollers 2 to form a nip 8.

In use, a plaster slurry 9 and the pre-generated foam mix 10, detailed above, are supplied under gravity, in an appropriate ratio, through conduits 4 and 5, respectively, into mixing box 3 where they are mixed to form a foamed plaster mixture 11. The foamed plaster mixture is then sprayed through the plurality of nozzles 6,6',6" onto a lower paper liner 12 which is being continuously conveyed, in the direction indicated, by rollers 2. The foamed plaster mixture becomes substantially evenly distributed across, and adheres to, the paper liner. An upper paper liner 13 is continuously conveyed, in the direction indicated by roller 7 into nip 8 where buildup of, the foamed plaster mixture into a dam 9 occurs and the plaster mixture adheres to the paper 13. Uncured plasterboard 14 is continuously conveyed downstream from the forming



station along a long conveyor belt allowing the chemical reactions of setting to take place. It is then cut to the required length; and dried by passage through multideck drying zones.

- 5 In an alternative process, which is not illustrated, a non-foamed slurry is poured into block shaped moulds, in which it is allowed to set partially. The blocks are hard enough to handle after a few minutes, at which time they are pushed from the mould using, for instance, a hydraulic
10 jack, and are conveyed to an oven to complete the setting/drying process.



CLAIMS

1. A process for producing a gypsum product involving the following steps:

- a) a slurry of gypsum is formed in water
- 5 b) the slurry is introduced to mould means and the gypsum allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an
10 emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at
15 least 50; and
- ii) an anionic dispersant which is a sulphated compound.

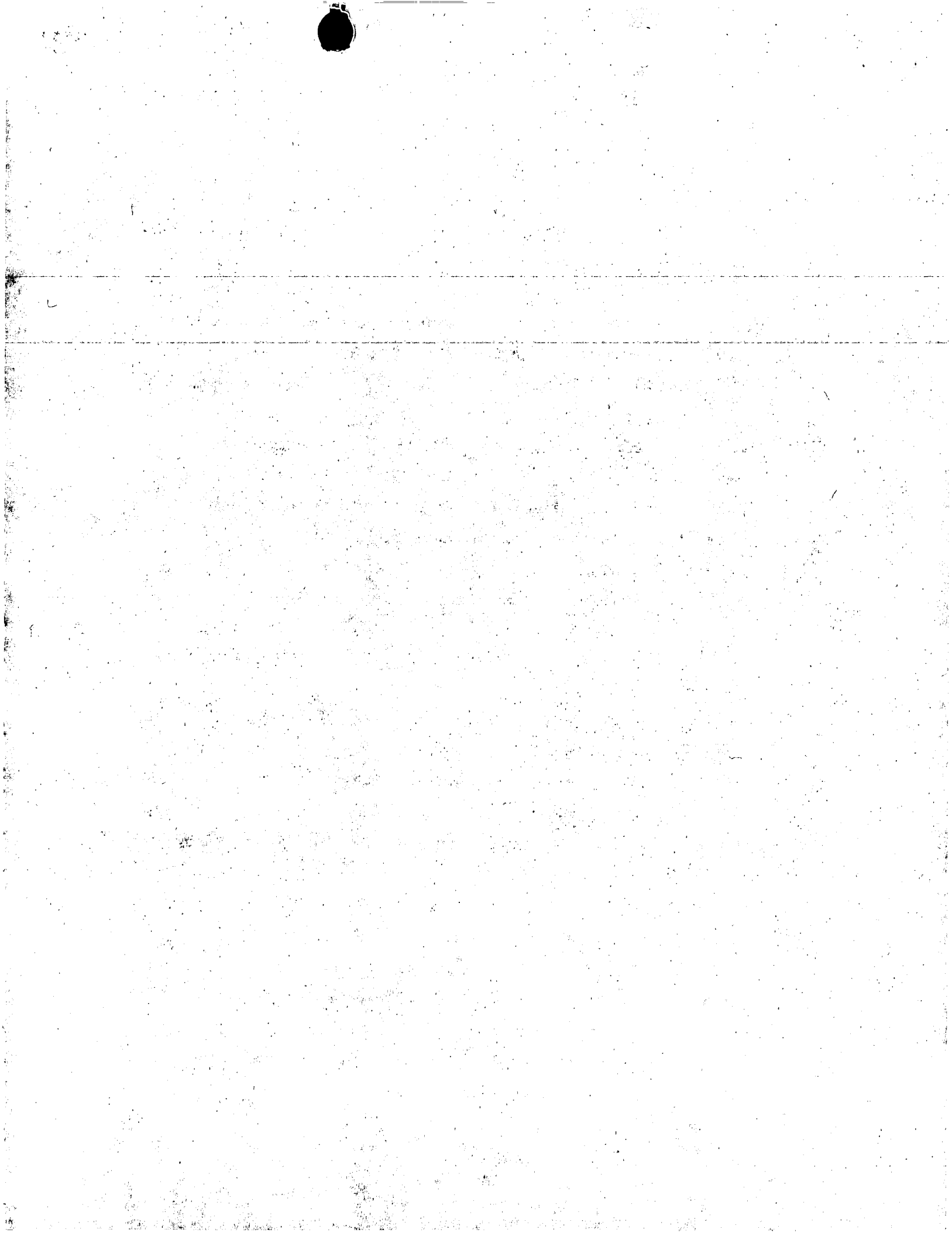
2. A process according to claim 1 in which the process is continuous and involves pouring of the slurry
20 onto a continuously moving belt.

3. A process according to claim 1 or 2 in which the gypsum slurry is foamed before introduction into the mould means.

4. A process according to claim 1 in which the
25 anionic dispersant is a sulphate or a sulphonate.

5. A process according to claim 4 in which the anionic dispersant is a polymeric compound, preferably an aryl sulphonate.

6. A process according to claim 5 in which the
30 anionic dispersant is a naphthalene sulphonate, preferably the sodium salt.



7. A process according to any preceding claim in which the nonionic surfactant is a higher alkanol, alkenol, alkanolic or alkenolic acid or aryl alcohol (including phenol) or carboxylic acid ethoxylated with at least 2
5 equivalents of ethylene oxide, preferably up to 100, for instance 3 to 30 equivalents, ethylene oxide.

8. A process according to claim 7 in which the nonionic surfactant is a C₈₋₁₈-alkanol or -alkenol ethoxylated with 3 to 30 moles of ethylene oxide.

10 9. A process according to any preceding claim in which the petroleum-derived hydrocarbon wax (a) is one with a high melting point and a low oil content, preferably a paraffin wax, more preferably such a wax having a
congealing point in the range 55 to 69°C (ASTM D938) a
15 penetration value (by ASTM D1321) at 25°C in the range 10 to 20 mm/10 and at 50°C at least 50 mm/10 and a viscosity at 100°C (by ASTM D445) in the range 3-7 cSt.

10. A process according to any preceding claim in which the montan wax has a congealing point in the range
20 67-80°C, an acid value (by ASTM D1980) in the range 10 to 37 mgKOH/g, a saponification value (by ASTM D1962) in the range 35 to 100 mgKOH/g, a viscosity (by ASTM D445) at 90°C in the range 20-400 cSt and at 100°C in the range 20 to 200 cSt.

25 11. A process according to any preceding claim in which, in the emulsifier, the montan wax is present in an amount in the range 10 to 20% by weight, the hydrocarbon wax is present in an amount in the range 20 to 40% by weight and the emulsifier system is present in an amount in
30 the range 1 to 6% by weight.

12. A process according to any preceding claim in which the ratio of the anionic dispersant to nonionic

surfactant in the emulsion is in the range 5:1 to 1:5, preferably 3:1 to 1:3.

13. A process according to any preceding claim in which the emulsion is added to the gypsum slurry in an amount in the range 0.5 to 10%, preferably in the range 1.0 to 5.0% by weight based on the weight of gypsum.

14. A process according to any preceding claim in which the mould means has a paper liner which becomes permanently laminated to the solidified gypsum.

15. A process according to any preceding claim in which the water in which the gypsum slurry is formed has a hardness of at least 100 ppm Ca^{2+} , preferably at least 150 ppm Ca^{2+} , more preferably at least 200 ppm Ca^{2+} .

16. An emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.

17. An emulsion according to claim 16 having the further features defined in any of claims 2 to 12.

18. A method of forming an emulsion in which a petroleum derived hydrocarbon wax and a montan wax are each melted and blended in molten form, an emulsifier system is dissolved into water to form an aqueous emulsifier solution and the molten wax mixture is dispersed into the aqueous emulsifier solution to form an emulsion, characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.

19. A method according to claim 18 in which the emulsifier system is as defined in any of claims 2 to 8 and 12 and/or the waxes are as defined in claim 9 and/or claim 10.

20. A method according to claim 18 or 19 in which the montan wax is used in an amount in the range 10 to 20% by weight of the emulsion, the hydrocarbon wax is used in an amount in the range 20 to 40% by weight of the emulsion and the emulsifier system is used in an amount in the range 0.5 to 6% by weight, preferably 1 to 2.5% by weight of the emulsion.

1/2

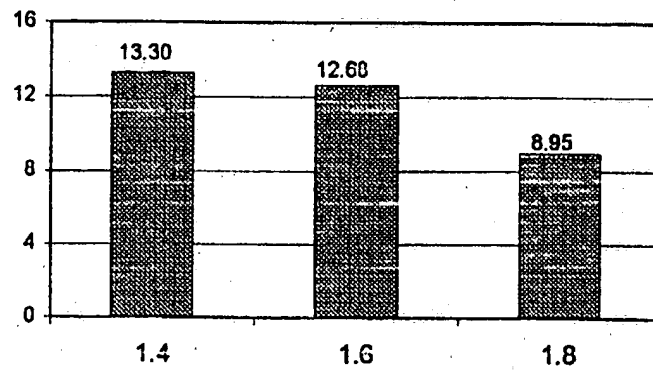


Figure 1

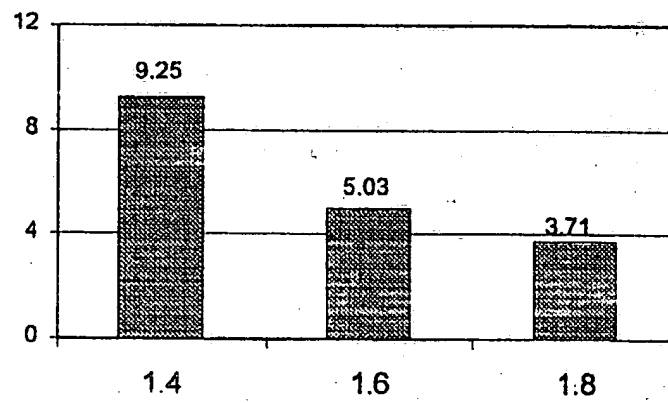


Figure 2

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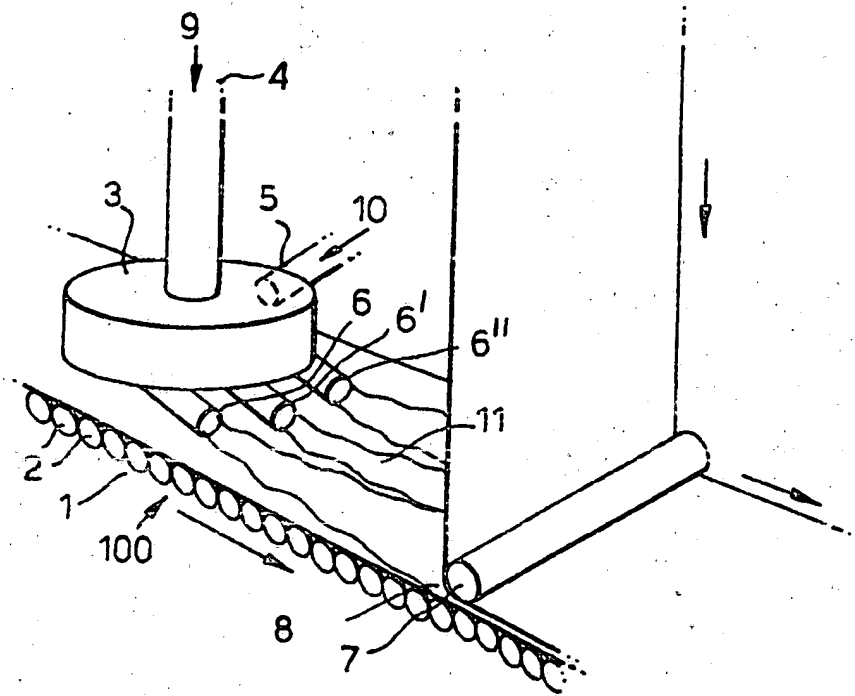


Figure 3

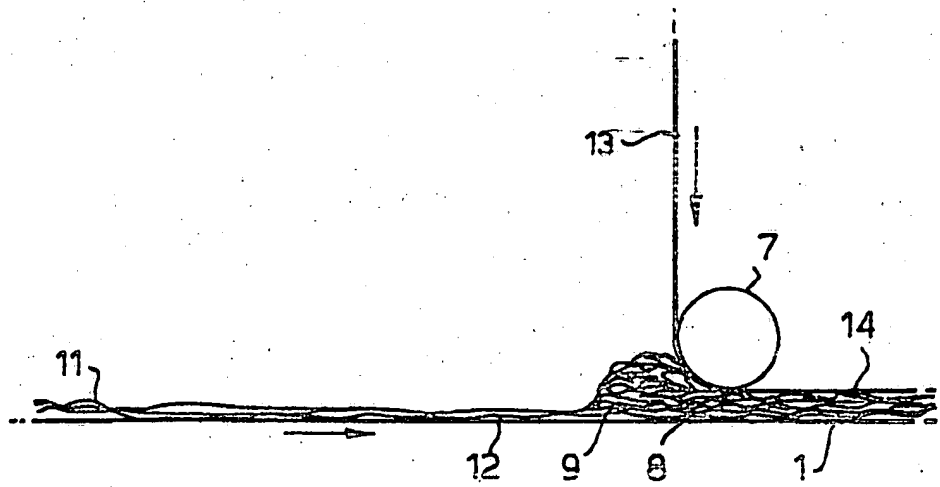


Figure 4

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/00064

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C04B28/14 C08L91/06 //(C04B28/14, 24:08, 24:22, 24:32, 24:36),
C04B111:27

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C04B C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 695 553 A (T. CLARET, ET AL.) 9 December 1997 see claim 1	1,4,9, 11,13, 14,16-20
A,P	WO 98 09925 A (MOBIL OIL) 12 March 1998 cited in the application see page 4, line 1-11; claims 1,8	1,3-6
A	US 4 315 957 A (HOECHST AG) 16 February 1982 see column 3, line 6-10 see column 3, line 35-45; claim 1	16,18,20
	-/--	

☒ Further documents are listed in the continuation of box C.

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Date of the actual completion of the international search

22 April 1999

Date of mailing of the international search report

03/05/1999

Name and mailing address of the ISA

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Fax: (+31-70) 340-3016

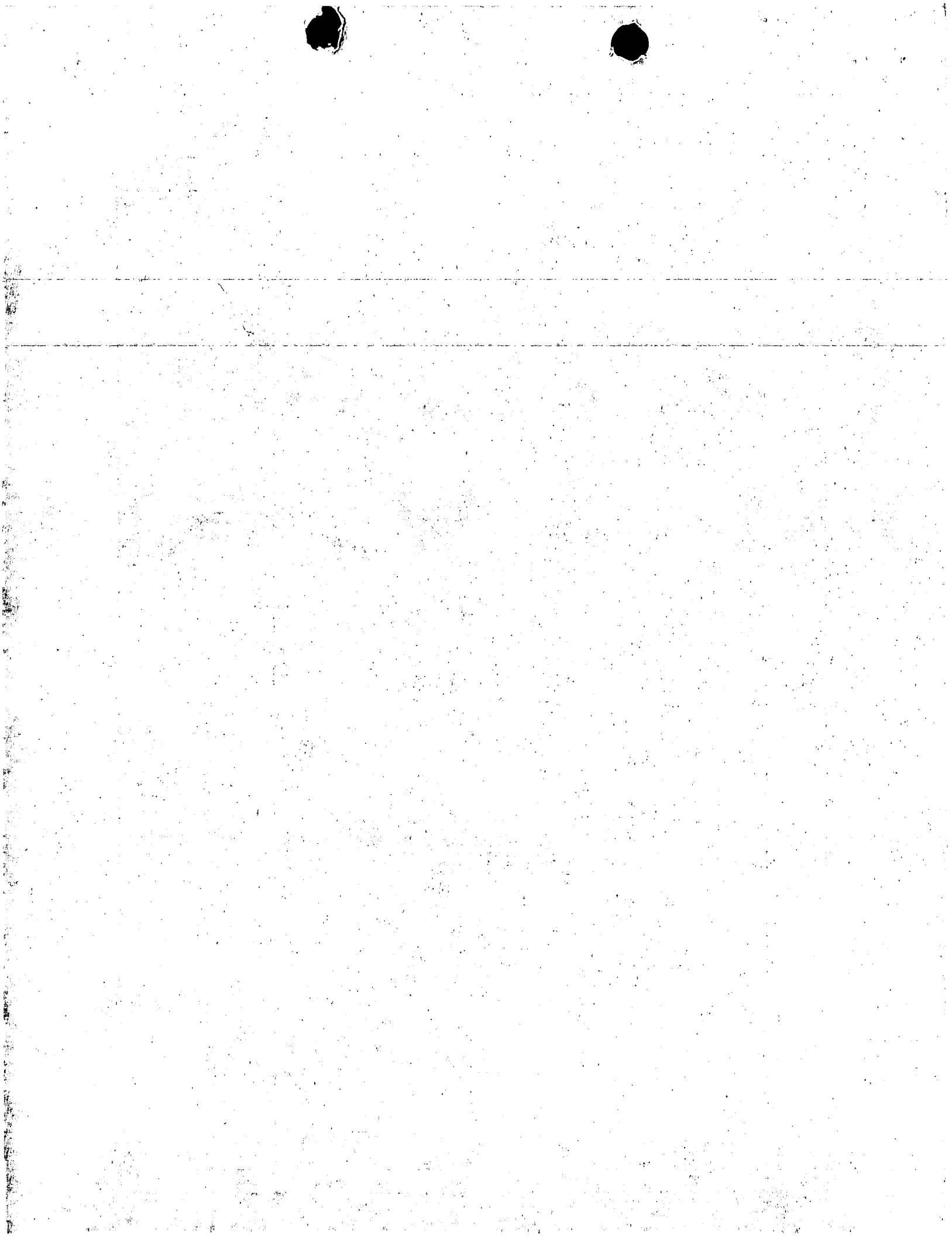
Authorized officer

Daeleman, P

INTERNATIONAL SEARCH REPORT

International Application No
PCT/GB 99/00064

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A,P	PATENT ABSTRACTS OF JAPAN vol. 098, no. 008, 30 June 1998 & JP 10 067980 A (MITSUBISHI CHEM CORP), 10 March 1998 see abstract	16
A	US 5 437 722 A (L. BORENSTEIN) 1 August 1995 cited in the application	
A	DATABASE WPI Week 8635 Derwent Publications Ltd., London, GB; AN 86-229176 XP002100852 & JP 61 159494 A (SANWA KAGAKU KK) see abstract	



INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 99/00064

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5695553 A	09-12-1997	CA 2184041 A	24-02-1998
WO 9809925 A	12-03-1998	AU 4125397 A	26-03-1998
US 4315957 A	16-02-1982	DE 2926197 A	15-01-1981
		AU 535960 B	12-04-1984
		AU 5972480 A	08-01-1981
		BR 8004068 A	21-01-1981
		EP 0022212 A	14-01-1981
		JP 56010559 A	03-02-1981
		JP 63058193 B	15-11-1988
		ZA 8003853 A	29-07-1981
US 5437722 A	01-08-1995	AU 676965 B	27-03-1997
		CA 2116483 A,C	26-08-1995
		EP 0669377 A	30-08-1995
		JP 8337459 A	24-12-1996
		AT 175707 T	15-01-1997
		AU 1637595 A	24-10-1996
		DE 69507174 D	25-02-1999

INTERNATIONAL COOPERATION TREATY

PTO/PCT Rec'd 07 JUL 2000

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PCT

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THE INTERNATIONAL SEARCH REPORT
OR THE DECLARATION

(PCT Rule 44.1)

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03/05/1999

Applicant's or agent's file reference

HMJ03045W0

FOR FURTHER ACTION

See paragraphs 1 and 4 below

International application No.

PCT/GB 99/00064

International filing date
(day/month/year)

08/01/1999

Applicant

MOBIL OIL COMPANY LIMITED et al.

1. ☒ The applicant is hereby notified that the International Search Report has been established and is transmitted herewith.

Filing of amendments and statement under Article 19:

The applicant is entitled, if he so wishes, to amend the claims of the International Application (see Rule 46):

When? The time limit for filing such amendments is normally 2 months from the date of transmittal of the International Search Report; however, for more details, see the notes on the accompanying sheet.

Where? Directly to the International Bureau of WIPO
34, chemin des Colombettes
1211 Geneva 20, Switzerland
Facsimile No.: (41-22) 740.14.35

For more detailed instructions, see the notes on the accompanying sheet.

2. ☐ The applicant is hereby notified that no International Search Report will be established and that the declaration under Article 17(2)(a) to that effect is transmitted herewith.

3. ☐ With regard to the protest against payment of (an) additional fee(s) under Rule 40.2, the applicant is notified that:

☐ the protest together with the decision thereon has been transmitted to the International Bureau together with the applicant's request to forward the texts of both the protest and the decision thereon to the designated Offices.

☐ no decision has been made yet on the protest; the applicant will be notified as soon as a decision is made.

4. **Further action(s):** The applicant is reminded of the following:

Shortly after 18 months from the priority date, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau as provided in Rules 90bis.1 and 90bis.3, respectively, before the completion of the technical preparations for international publication.

Within 19 months from the priority date, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase until 30 months from the priority date (in some Offices even later).

Within 20 months from the priority date, the applicant must perform the prescribed acts for entry into the national phase before all designated Offices which have not been elected in the demand or in a later election within 19 months from the priority date or could not be elected because they are not bound by Chapter II.

Name and mailing address of the International Searching Authority



European Patent Office, P.B. 5818 Patentaan 2
NL-2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Alex Schmidt

PATENT COOPERATION TREATY

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INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference HMJ03045W0	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/GB 99/ 00064	International filing date (day/month/year) 08/01/1999	(Earliest) Priority Date (day/month/year) 08/01/1998
Applicant MOBIL OIL COMPANY LIMITED et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. Basis of the report

a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.

☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).

b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing :

☐ contained in the international application in written form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished

2. ☐ **Certain claims were found unsearchable** (See Box I).

3. ☐ **Unity of invention is lacking** (see Box II).

4. With regard to the title,

☒ the text is approved as submitted by the applicant.

☐ the text has been established by this Authority to read as follows:

5. With regard to the abstract,

☒ the text is approved as submitted by the applicant.

☐ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

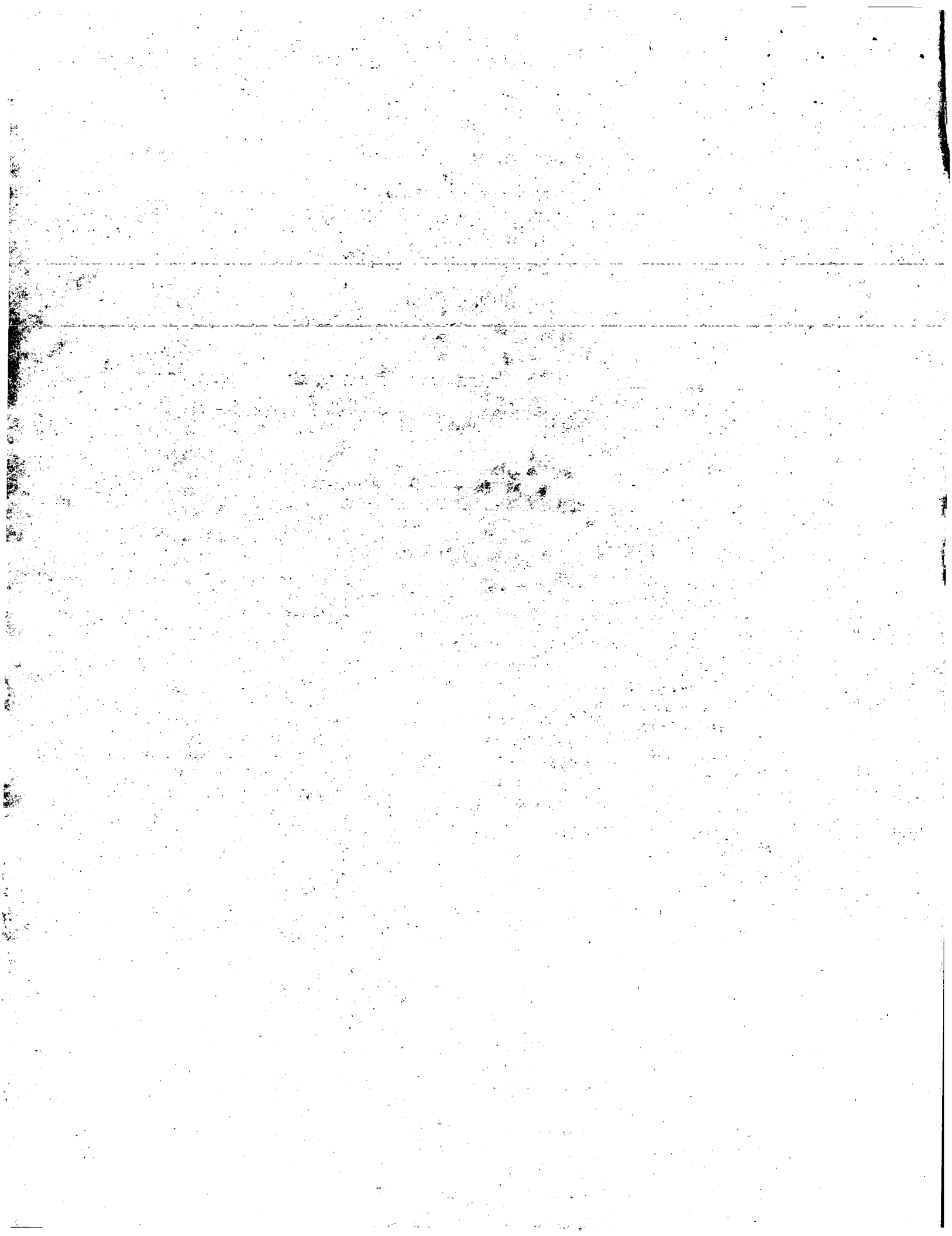
6. The figure of the drawings to be published with the abstract is Figure No.

☐ as suggested by the applicant.

☐ because the applicant failed to suggest a figure .

☐ because this figure better characterizes the invention.

None of the figures.



INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/00064

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C04B28/14 C08L91/06 //(C04B28/14, 24:08, 24:22, 24:32, 24:36),
C04B111:27

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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C04B C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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A,P	WO 98 09925 A (MOBIL OIL) 12 March 1998 cited in the application see page 4, line 1-11; claims 1,8	1,3-6
A	US 4 315 957 A (HOECHST AG) 16 February 1982 see column 3, line 6-10 see column 3, line 35-45; claim 1 -/-	16,18,20

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Daeleman, P

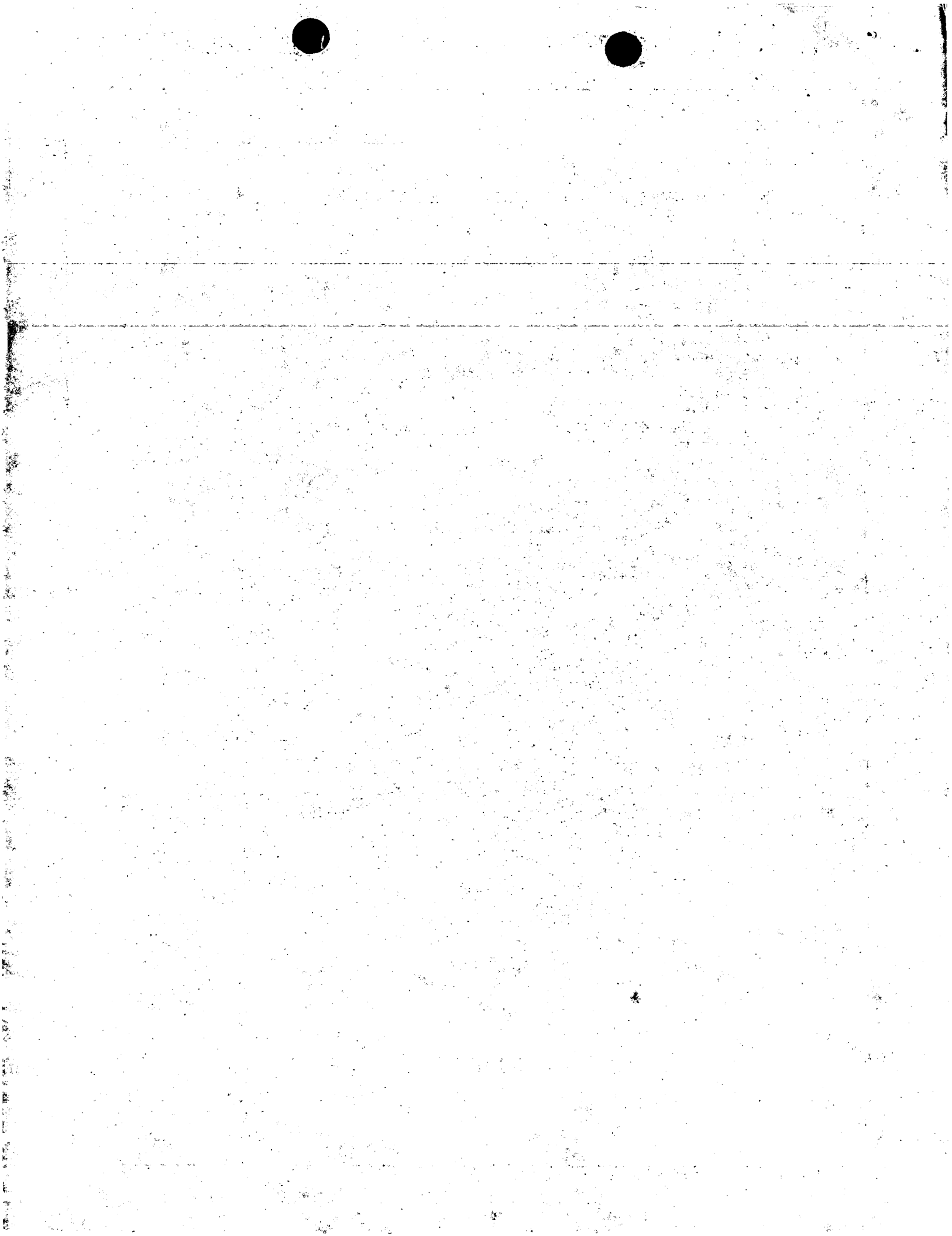
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International Application No

PCT/GB 99/00064

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Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A, P	<p>PATENT ABSTRACTS OF JAPAN vol. 098, no. 008, 30 June 1998 & JP 10 067980 A (MITSUBISHI CHEM CORP), 10 March 1998 see abstract</p> <p>---</p>	16
A	<p>US 5 437 722 A (L. BORENSTEIN) 1 August 1995 cited in the application</p> <p>---</p>	
A	<p>DATABASE WPI Week 8635 Derwent Publications Ltd., London, GB; AN 86-229176 XP002100852 & JP 61 159494 A (SANWA KAGAKU KK) see abstract</p> <p>-----</p>	



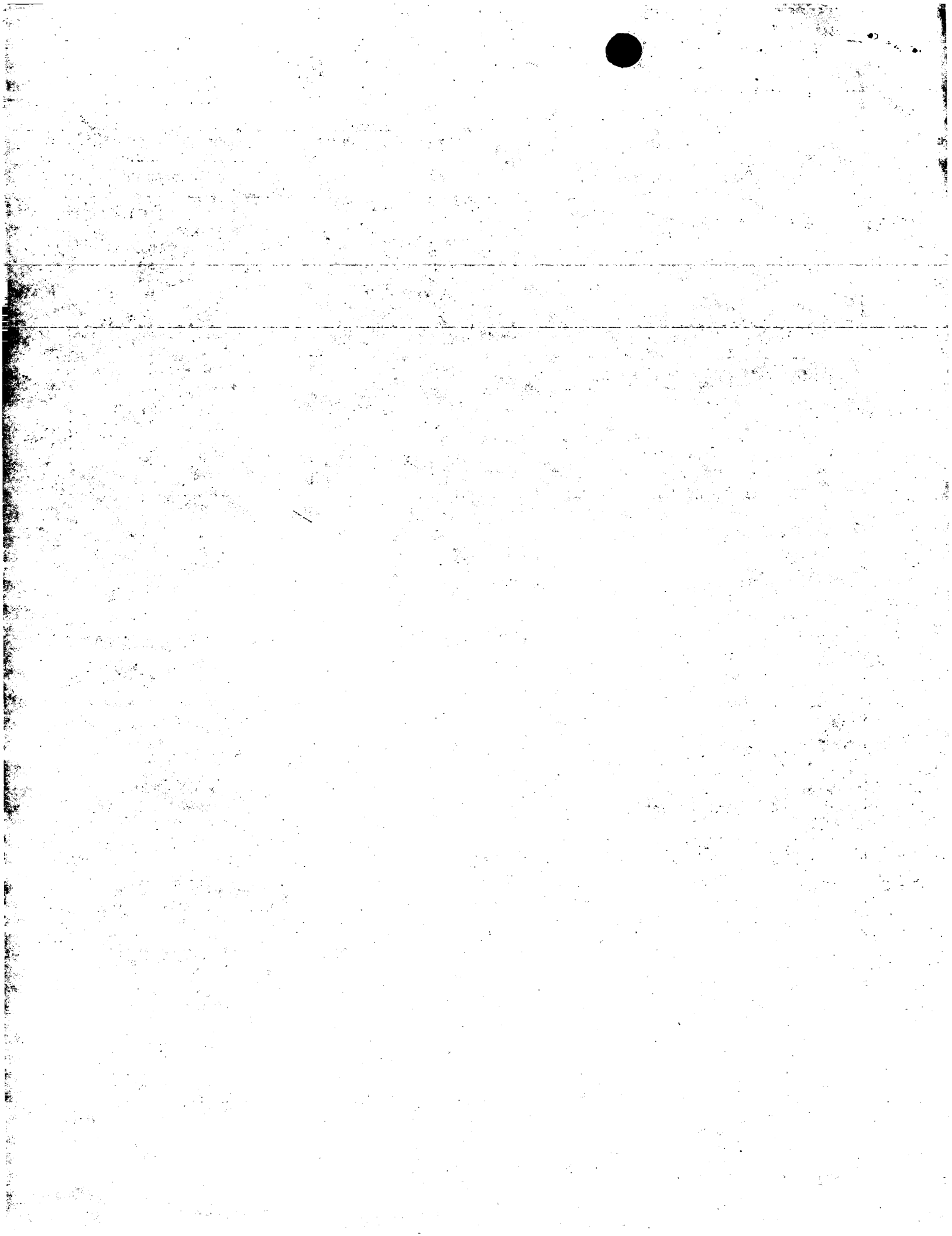
INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 99/00064

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 5695553	A	09-12-1997	CA	2184041 A	24-02-1998
WO 9809925	A	12-03-1998	AU	4125397 A	26-03-1998
US 4315957	A	16-02-1982	DE	2926197 A	15-01-1981
			AU	535960 B	12-04-1984
			AU	5972480 A	08-01-1981
			BR	8004068 A	21-01-1981
			EP	0022212 A	14-01-1981
			JP	56010559 A	03-02-1981
			JP	63058193 B	15-11-1988
			ZA	8003853 A	29-07-1981
US 5437722	A	01-08-1995	AU	676965 B	27-03-1997
			CA	2116483 A,C	26-08-1995
			EP	0669377 A	30-08-1995
			JP	8337459 A	24-12-1996
			AT	175707 T	15-01-1997
			AU	1637595 A	24-10-1996
			DE	69507174 D	25-02-1999



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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C04B 28/14, C08L 91/06 // (C04B 28/14, 24:08, 24:22, 24:32, 24:36), 111:27		A1	(11) International Publication Number: WO 99/35103
			(43) International Publication Date: 15 July 1999 (15.07.99)
(21) International Application Number: PCT/GB99/00064 (22) International Filing Date: 8 January 1999 (08.01.99) (30) Priority Data: 9800368.4 8 January 1998 (08.01.98) GB (71) Applicant (for all designated States except US): MOBIL OIL COMPANY LIMITED [GB/GB]; Mobil House, 500-600 Witan Gate, Central Milton Keynes, Buckinghamshire MK9 1ES (GB). (72) Inventor; and (75) Inventor/Applicant (for US only): BOELEE, Scotia [NZ/GB]; 105 Forest Road, Tunbridge Wells, Kent TN2 5BG (GB). (74) Agent: GILL JENNINGS & EVERY; Broadgate House, 7 Eldon Street, London EC2M 7LH (GB).			(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: GYPSUM PRODUCT			
(57) Abstract A wax emulsion comprising an emulsifying system containing a sulphated anionic surfactant and a non ionic surfactant having high water solubility (cloud point) and high foaming ability is added to a gypsum slurry to improve the moisture resistance of gypsum board. The wax is a mixture of a petroleum derived hydrocarbon wax and a montan wax.			

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CZ	Czech Republic	LC	Saint Lucia	RU	Russian Federation		
DE	Germany	LI	Licchtenstein	SD	Sudan		
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534 Rec'd PCT/PT 07 JUL 2000

GYPSUM PRODUCT

This invention relates to a gypsum product and to a process for its manufacture. More particularly, this invention relates to a foamed gypsum product of improved
5 water resistance and/or reduced density and to a process, preferably to a continuous process, for its manufacture.

Gypsum board (or plaster board or wallboard) is used extensively in the construction industry. It typically comprises a substantially flat core of set gypsum on either
10 side of which a liner may be adhered. A liner typically comprises paper. The core may be reinforced; for example, reinforced with glass fibres.

Gypsum products (or Plaster of Paris or plaster products) are produced by mixing anhydrous calcium sulphate or calcium sulphate hemihydrate with water, and permitting
15 the mixture to set thereby producing calcium sulphate dihydrate. Often the slurry is foamed by incorporating a preformed solution of foaming agent in water (a surface active material) before adding to the mould means. A
20 pervasive problem with gypsum products, however, is that calcium sulphate dihydrate absorbs water and this reduces the strength of the gypsum product. Because of this, plaster board (for example) is required, at least in uses where a relatively high humidity is anticipated (for
25 example, kitchens or bathrooms) to be substantially moisture resistant and this requires the presence of a hydrophobing agent. ("Hydrophobing" is a term used in the art to denote a method of preventing, or reducing water absorption).

30 Silicone oil has previously been used as a hydrophobing agent for gypsum products. It is, however, expensive and in relatively short supply. It also has

difficulty in providing a moisture resistance of less than 5 wt % water absorption in the test hereinafter mentioned.

In US-A-5437722 an aqueous emulsion comprising a hydrocarbon wax, a montan wax and emulsifier/stabiliser system and also including a polyvinyl alcohol, is used to render gypsum products water resistant. The emulsifier system may include non-ionic or anionic surfactant and alkali. Examples of non ionic surfactants are alkyl-phenoxypoly(ethyleneoxy) ethanols, sorbitan fatty acid esters and polyoxyethylene sorbitan fatty acid esters. Examples of anionic surfactants are saponified fatty acids.

In our copending application number PCT/GB97/02366, unpublished at the priority date of the present invention, we describe a foamed gypsum product which is hydrophobed by incorporation of an aqueous emulsion comprising a hydrocarbon wax, a montan wax and a colloid stabilised emulsifier system. The preferred colloid stabilised emulsifier system comprises either organic or, more preferably, inorganic colloidal materials. One example is a montmorillonite clay based system in combination with a sodium naphthalene sulphonate.

In the above mentioned PCT application, a comparative example uses an emulsifier system based on a combination of a nonionic surfactant, which was not specifically defined, with an anionic surfactant again, not specifically defined. The emulsifier system used in that comparative example was in fact a combination of an alkyl phenyl ethoxylate with a soap-type anionic surfactant. The worked example using that emulsifier system did not work. In fact it is now believed that the anionic surfactant caused collapse of the foam or that, upon addition to a slurry formed using

relatively hard water, the anionic surfactant was precipitated out of the system by the hardness ions.

The above mentioned PCT application also discloses a comparative example using a nonionic surfactant based emulsifier system including no anionic emulsifier. Whilst this gave some improvement over the mixed system, it was found that using a high enough level of emulsion in the gypsum product to achieve adequate density and/or water adsorption figures lead to over-wetting of paper used for the gypsum product and delamination during production.

In the present invention there is provided a process for producing a foamed gypsum product involving the following steps:

- a) a slurry of gypsum is formed in water
- b) the slurry is introduced to mould means and allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point (in saline per DIN 53917) of at least 50; and
- ii) an anionic dispersing agent which is a sulphated compound.

Preferably the anionic dispersant is a so called acid stable compound, that is the compound is ionised over a wide range of pH's including acidic pH. The acid stability can be judged by the pK_a of the conjugate acid, which should preferably be less than 4, more preferably less than

3, for instance 2 or less. The anionic dispersant is generally a sulphate or a sulphonate.

A suitable class of anionic dispersants are sulphated naphthalene/formaldehyde condensates, for instance having
5 molecular weight in the range 6000 to 40000. These compounds are also known as naphthalene sulphonates. Other aryl sulphonates may also be used. The anionic dispersant is generally used in the emulsion in the form of its sodium salt. Alternatively potassium, ammonium, or even divalent
10 metal salts such as calcium or magnesium, may be used. Suitable compounds are available from BASF AG under the trade name Tamol (trademark).

The nonionic surfactant must be relatively water soluble. The water solubility of non ionic surfactants can
15 be determined by standard test method DIN 53917 in saline. The component should have a cloud point of at least 50, for instance more than 60, up to around 100, for instance approximately 75.

We have found that the nonionic surfactant giving
20 optimum performance is one which has a high foaming ability. Foaming ability can be measured by standard test methods DIN 53902. For instance the test should be carried out according to the method given in sheet 1 of that standard test method, at 40°C, with the surfactant being
25 used in a concentration of 2 g/l in water containing 1.8 mmol Ca ions/l, the duration of the test being 30 seconds. The foaming ability should be at least 300, more preferably at least 500, for instance up to 750. Nonionic surfactants with foaming ability using the above mentioned test method
30 of around 600 are available.

The nonionic surfactant is generally an ethoxylated higher alkyl, alkenyl, alkanoyl or alkenoyl compound.

Alternatively ethoxylated aryl compounds may be used, for instance ethoxylated alkyl phenol derivatives. Preferably the compound is a C₈₋₁₈-alkanol ethoxylated with 3 to 30 equivalents of ethylene oxide, for instance a C₁₂₋₁₈-alkanol ethoxylated with 10 to 20 equivalents of ethylene oxide. Suitable compounds are available under the trade name Lutensol (trademark) from BASF AG. It may alternatively be possible to use polyglycosylated alkyl, alkenyl, alkanoyl, alkenoyl and aryl compounds for instance alkyl polyglucosides.

The use of an acid stable anionic dispersant, it is believed, avoids the addition of the emulsion resulting in the collapse of the foam in the gypsum slurry. Accordingly the density of the product is optimised. The use of the anionic dispersant in combination with non ionic surfactant avoids the use of such high concentrations of nonionic dispersant in the gypsum slurry which can lead to over-wetting of paper used in the gypsum product and delamination during production. The preferred surfactant makes the emulsion suitable for use with gypsum slurries made up in hard water, for instance water having hardness value of at least 100 ppm Ca²⁺ even more than 150 ppm Ca²⁺, for instance at least 200 ppm Ca²⁺.

The use of the high foaming non ionic surfactant is believed also to contribute to optimisation of the gypsum slurry foam and the density of the final product. The preferred combination of surfactants in the emulsifier system allows high levels of wax to be incorporated into the final product for optimum hydrophobing of the gypsum product.

The emulsifier system has process advantages also in production of non-foamed products, for instance, other

products made on continuous lines such as fire resistance board. The system provides good compatibility with the equipment

The petroleum-derived hydrocarbon wax (a) is preferably one with a high melting point and a low oil content. A preferred such wax is a paraffin wax, such as fully refined paraffin wax. Fully refined paraffin waxes are generally obtained from highly paraffinic refinery streams such as those obtained from the solvent dewaxing of distillates and other lube fractions. The product is further typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION	
		MIN	MAX
15 Congealing Point (°C)	ASTM D938	55	69
Oil in Wax (%)	ASTM D721		1
Penetration at 25°C (mm/10)	ASTM D1321	10	20
20 Penetration at 50°C (mm/10)	ASTM D1321		80
Viscosity (cSt @ 100°C)	ASTM D445	3	7

25 An example of a fully refined paraffin wax which has been found to be entirely satisfactory, and which satisfies the above specification, is MOBILWAX 135 (derived from the 150 SPN stream) as supplied by Mobil Oil Company Limited; MOBILWAX 145 or 150 (derived from the 300 or 450 SPN stream) are also suitable. While these waxes are hydrofinished to give a white colour and good odour, unfinished wax (which differs only in colour and odour) is also suitable for use in accordance with the invention.

The petroleum-derived hydrocarbon wax a) suitably comprises from 20 to 40 wt % of the aqueous emulsion, preferably from 25 to 35 wt % of the aqueous emulsion.

The montan wax or lignite wax b) is another wax with a high melting point. It is preferably used in crude (or raw) form. Such a product is typically characterised as follows:

CHARACTERISTICS	TEST METHOD	SPECIFICATION		PREFERRED SPECIFICATION	
		MIN	MAX	MIN	MAX
Congealing Point (°C)	ASTM D938	67	80	75	80
Acid Value (mg KOH/g)	ASTM D1980	10	37	10	20
Saponification Value (mgKOH/g)	ASTM D1962	35	100	65	90
Ash Content (% wt)	ASTM D482		1		1.0
Density at 20°C (g/cm ³)	ASTM D1298	0.95	1.04	0.95	1.04
Viscosity (cSt at 90°C)	ASTM D445	20	400	150	400
Viscosity (cSt at 100°C)	ASTM D445	20	200	60	150

The montan wax b) suitably comprises from 10 to 20 wt % of the aqueous emulsion, preferably from 11 to 15 wt % of the aqueous emulsion.

The emulsifier system (i.e. the total of two or more components of a multi-component system) is suitably present in an amount from 0.5 to 6 wt %, preferably 1.2 to 5 wt %, more preferably 1.5 to 4% of the aqueous emulsion. The

ratio of the non-ionic and anionic components is preferably in the range 5:1 to 1:5, more preferably 3:1 to 1:3, most preferably 2:1 to 1:2.

In the invention the slurry in water, preferably
5 contains 100 parts by weight of gypsum and from 0.5 to 10, preferably from 1 to 5 % by weight of an emulsion as herein defined. The slurry suitably contains 50-60 weight % gypsum and 40-50 weight % water, preferably about 55% gypsum. An accelerator is usually added, for instance a
10 slurry mix from a previous batch.

The slurry preferably contains a foaming agent. Preferably the product is a paper lined board and the process thus preferably includes a step of foaming, usually involving formation of a pre-formed foam by vigorous
15 stirring of the foaming agent in water, followed by mixing the prefoam into preformed gypsum slurry containing the emulsion.

This invention also provides a water-resistant gypsum product which comprises the set composition. Whilst the
20 product may be an unlined board, the invention is particularly applicable to a product which comprises a core product of a set such composition sandwiched between a pair of liners usually paper liners. Another suitable product is fire resistant board which has a glass fibre scrim
25 embedded in each surface of the gypsum board, which is generally unfoamed. The invention includes also the emulsion itself and the process for making it.

The invention further provides a process for the preparation of a water-resistant gypsum board product,
30 which process comprises forming a mixture which is a slurry in water containing 100 parts by weight of gypsum and from 0.5 to 5 parts by weight of an emulsion according to the

invention; forming a layer of the mixture in a mould means and drying the layer of gypsum mixture, while permitting hydration of the gypsum, for form a board product. Preferably the process is continuous. The process may be
5 for forming tiles or blocks or boards. Blocks may be formed in moulds from which they are removed when set. Tiles or boards may be formed by spreading a layer of the gypsum mixture on a first planar substrate, a second planar substrate is positioned over the layer to form an assembly,
10 and the mixture is allowed to set in the assembly. A gasket may be provided between the planar substrates. Where the product is a lined board, the first and second planar substrates are constituted by liner, for instance paper, usually supported in a mould. Where the product is
15 to be unlined, the planar substrates are removed when the product is set. Where the product is a block, it is usually removed from a mould before the mixture is completely set, but when it is hard enough to handle. Where the product is a fire resistant board a fibreglass
20 scrim is embedded in each side of the slurry in the mould means by feeding to each side of the poured (unfoamed) slurry before the nip forming a dam in an apparatus similar to that shown in Figures 3 and 4 below.

This invention includes the use of an aqueous emulsion
25 of the invention to furnish a gypsum product with water resistance and the use of an aqueous emulsion of the invention to aid foaming of a gypsum slurry, for instance to reduce the density of the set foamed gypsum product.

Figure 1 shows the water absorption results of the
30 product of the comparative example.

Figure 2 shows the water absorption results of the product of the example of the invention.

Figures 3 and 4 are a schematic representations of a foaming station for lined gypsum board.

The following Example illustrate the invention.

EXAMPLE

5 First the wax phase was prepared by dissolving 12 wt % crude montan wax (Crude Montan Wax supplied by Schuemann Sabol GmbH) in 30 wt % of fully refined paraffin wax (MOBILWAX 135 supplied by Mobil Oil Company Limited) at a suitable raised temperature. 1% by weight sodium
10 naphthalene sulphonate (a sulphated naphthalene formaldehyde condensate having a molecular weight of about 6000 to 40000) from the TAMOL (trademark) range supplied by BASF was added to water to form the aqueous phase and stirred for a period. 0.45% Non ionic surfactant (added as
15 a 90% aqueous solution) (a C₁₃ alkanol - 12 mole ethoxylate available as Lutensol TO12 series) was then added to the aqueous phase and stirred for a period. The temperature of the wax melt was lowered to 100°C and the wax phase was next added to the aqueous phase heated to a suitable
20 temperature, with stirring for a suitable period to form a pre-emulsion. The pre-emulsion still at a raised temperature was next recycled through a homogeniser, with no impressed pressure, for a full pass. Gradually, the pressure was increased to a value in the range 20-25 MPa
25 (220 bar) and the emulsion recycled for a further pass to form an aqueous emulsion in accordance with the invention.

The emulsion was then tested for its performance in the production of a gypsum product. A conventional foaming agent was mixed with vigorous stirring with a suitable
30 quantity water to generate a foam mixture. A gypsum slurry mix was prepared by adding a predetermined amount (1.4, 1.6 or 1.8% by weight based on the amount of gypsum) of wax

emulsion (according to the invention or comparative) to around 40 parts by weight water along with predetermined amounts of a wetting agent, starch and an accelerator in a total amount of 0.38 parts by weight. To this around 58 parts by weight gypsum was added with stirring. The pre-generated foam mix was next added to the gypsum slurry and stirred to form a foamed gypsum slurry. The slurry was poured into a paper lined mould of 300 x 300 x 12.5 mm dimension and a second sheet of paper placed on top to form a gypsum coupon which was then dried in three stages of successively lower temperatures and longer times to a constant weight. The density and 2 hour water absorption were then determined. The density was calculated by dividing the dry weight of the test specimen by the mould volume. The water absorption was determined by cutting a test specimen measuring 280 x 280 mm from the coupon and immersing this specimen in a water bath at 23°C covered with 25 to 35 mm of water for 2 hours. Its weight before and after immersion was measured and the percentage increase calculated.

The results, which include comparative tests, are shown in figures 1 and 2. In these tests density and 2 hr water absorption were measured and reported using the emulsifier system of the invention and also as comparison, based on the colloid emulsified system of the above mentioned PCT publication, in which the same amounts of wax emulsion containing an emulsifier system of a bentonite clay and sodium naphthalene sulphonate.

A further example has also been conducted in which the emulsion containing Mobilwax 135, which has a melting point (congealing point) in the range 57-60°C and a maximum content of oil of 1.0wt%, is used at a level of 4% by

weight. This example is then repeated, but using emulsions (at 4% by weight in the gypsum) in which the Mobil wax 135 is replaced by waxes having higher (63-66°C , and 66-69°C, respectively) and lower (54-57°C) melting/congealing points. When used at the same levels in the emulsion, the emulsion (at the same level in the gypsum) produced good results for water absorption. The values were less than 5%, indeed less than 2.5%, in each case.

In Figures 3 and 4 there is shown a forming station for gypsum board manufacture is shown generally at 100. It comprises a conveyor 1 which is formed from an array of like, generally coplanar, parallel driven rollers 2 which are rotatable in the same sense. Above the conveyor is a manifold mixing box 3 into which entry conduits 4 and 5 and a plurality of exit nozzles 6,6',6" are flowably connected. A contra-rotatable roller 7 is biased toward rollers 2 to form a nip 8.

In use, a plaster slurry 9 and the pre-generated foam mix 10, detailed above, are supplied under gravity, in an appropriate ratio, through conduits 4 and 5, respectively, into mixing box 3 where they are mixed to form a foamed plaster mixture 11. The foamed plaster mixture is then sprayed through the plurality of nozzles 6,6',6" onto a lower paper liner 12 which is being continuously conveyed, in the direction indicated, by rollers 2. The foamed plaster mixture becomes substantially evenly distributed across, and adheres to, the paper liner. An upper paper liner 13 is continuously conveyed, in the direction indicated by roller 7 into nip 8 where buildup of, the foamed plaster mixture into a dam 9 occurs and the plaster mixture adheres to the paper 13. Uncured plasterboard 14 is continuously conveyed downstream from the forming

station along a long conveyor belt allowing the chemical reactions of setting to take place. It is then cut to the required length; and dried by passage through multideck drying zones.

- 5 In an alternative process, which is not illustrated, a non-foamed slurry is poured into block shaped moulds, in which it is allowed to set partially. The blocks are hard enough to handle after a few minutes, at which time they are pushed from the mould using, for instance, a hydraulic
- 10 jack, and are conveyed to an oven to complete the setting/drying process.

CLAIMS

1. A process for producing a gypsum product involving the following steps:

a) a slurry of gypsum is formed in water

5 b) the slurry is introduced to mould means and the gypsum allowed to hydrate,

in which a hydrophobing agent comprising an emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an
10 emulsifier system is added to the slurry before introduction into the mould means, and is characterised in that the emulsifier system comprises:

i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at
15 least 50; and

ii) an anionic dispersant which is a sulphated compound.

2. A process according to claim 1 in which the process is continuous and involves pouring of the slurry
20 onto a continuously moving belt.

3. A process according to claim 1 or 2 in which the gypsum slurry is foamed before introduction into the mould means.

4. A process according to claim 1 in which the
25 anionic dispersant is a sulphate or a sulphonate.

5. A process according to claim 4 in which the anionic dispersant is a polymeric compound, preferably an aryl sulphonate.

6. A process according to claim 5 in which the
30 anionic dispersant is a naphthalene sulphonate, preferably the sodium salt.

7. A process according to any preceding claim in which the nonionic surfactant is a higher alkanol, alkenol, alkanoic or alkenoic acid or aryl alcohol (including phenol) or carboxylic acid ethoxylated with at least 2
5 equivalents of ethylene oxide, preferably up to 100, for instance 3 to 30 equivalents, ethylene oxide.

8. A process according to claim 7 in which the nonionic surfactant is a C₈₋₁₈-alkanol or -alkenol ethoxylated with 3 to 30 moles of ethylene oxide.

10 9. A process according to any preceding claim in which the petroleum-derived hydrocarbon wax (a) is one with a high melting point and a low oil content, preferably a paraffin wax, more preferably such a wax having a
congealing point in the range 55 to 69°C (ASTM D938) a
15 penetration value (by ASTM D1321) at 25°C in the range 10 to 20 mm/10 and at 50°C at least 50 mm/10 and a viscosity at 100°C (by ASTM D445) in the range 3-7 cSt.

10. A process according to any preceding claim in which the montan wax has a congealing point in the range
20 67-80°C, an acid value (by ASTM D1980) in the range 10 to 37 mgKOH/g, a saponification value (by ASTM D1962) in the range 35 to 100 mgKOH/g, a viscosity (by ASTM D445) at 90°C in the range 20-400 cSt and at 100°C in the range 20 to 200 cSt.

25 11. A process according to any preceding claim in which, in the emulsifier, the montan wax is present in an amount in the range 10 to 20% by weight, the hydrocarbon wax is present in an amount in the range 20 to 40% by weight and the emulsifier system is present in an amount in
30 the range 1 to 6% by weight.

12. A process according to any preceding claim in which the ratio of the anionic dispersant to nonionic

surfactant in the emulsion is in the range 5:1 to 1:5, preferably 3:1 to 1:3.

13. A process according to any preceding claim in which the emulsion is added to the gypsum slurry in an amount in the range 0.5 to 10%, preferably in the range 1.0 to 5.0% by weight based on the weight of gypsum.

14. A process according to any preceding claim in which the mould means has a paper liner which becomes permanently laminated to the solidified gypsum.

15. A process according to any preceding claim in which the water in which the gypsum slurry is formed has a hardness of at least 100 ppm Ca^{2+} , preferably at least 150 ppm Ca^{2+} , more preferably at least 200 ppm Ca^{2+} .

16. An emulsion of a mixture of a petroleum derived hydrocarbon wax and montan wax in an aqueous continuous phase containing an emulsifier system characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.

17. An emulsion according to claim 16 having the further features defined in any of claims 2 to 12.

18. A method of forming an emulsion in which a petroleum derived hydrocarbon wax and a montan wax are each melted and blended in molten form, an emulsifier system is dissolved into water to form an aqueous emulsifier solution and the molten wax mixture is dispersed into the aqueous emulsifier solution to form an emulsion, characterised in that the emulsifier system comprises:

- i) a nonionic surfactant characterised by a foaming ability of at least 300 and a cloud point of at least 50; and
- ii) an anionic dispersant which is a sulphated compound.

5

19. A method according to claim 18 in which the emulsifier system is as defined in any of claims 2 to 8 and 12 and/or the waxes are as defined in claim 9 and/or claim 10.

10

20. A method according to claim 18 or 19 in which the montan wax is used in an amount in the range 10 to 20% by weight of the emulsion, the hydrocarbon wax is used in an amount in the range 20 to 40% by weight of the emulsion and the emulsifier system is used in an amount in the range 0.5 to 6% by weight, preferably 1 to 2.5% by weight of the emulsion.

15

20

25

30

1/2

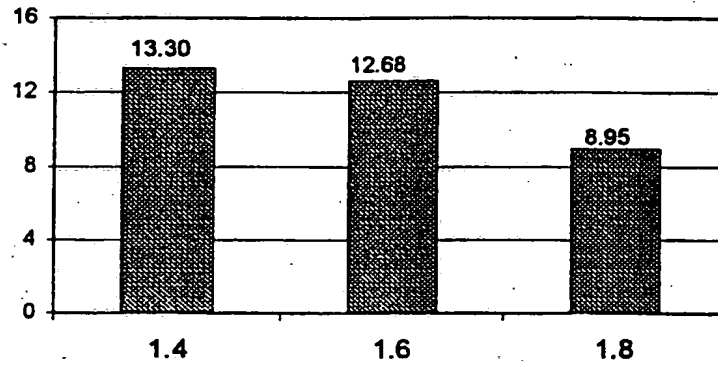


Figure 1

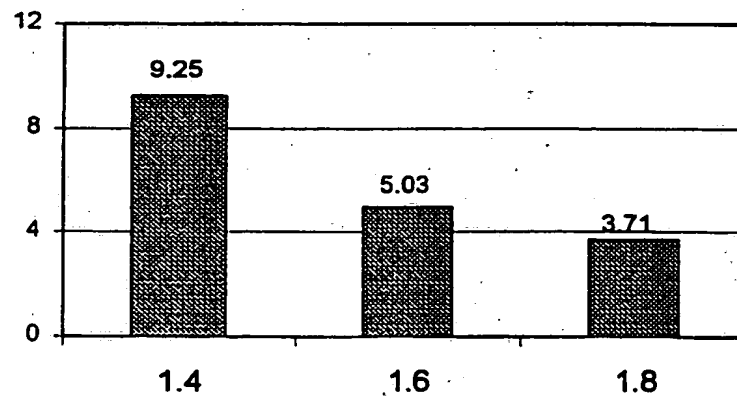


Figure 2

07 JUL 2000

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Figure 4

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 99/00064

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C04B28/14 C08L91/06 //(C04B28/14, 24:08, 24:22, 24:32, 24:36),
C04B111:27

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C04B C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5 695 553 A (T. CLARET, ET AL.) 9 December 1997 see claim 1	1, 4, 9, 11, 13, 14, 16-20
A, P	WO 98 09925 A (MOBIL OIL) 12 March 1998 cited in the application see page 4, line 1-11; claims 1, 8	1, 3-6
A	US 4 315 957 A (HOECHST AG) 16 February 1982 see column 3, line 6-10 see column 3, line 35-45; claim 1 -/--	16, 18, 20

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

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Date of the actual completion of the international search

22 April 1999

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Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Daeleman, P

INTERNATIONAL SEARCH REPORT

International Application No

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A, P	PATENT ABSTRACTS OF JAPAN vol. 098, no. 008, 30 June 1998 & JP 10 067980 A (MITSUBISHI CHEM CORP), 10 March 1998 see abstract ----	16
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Information on patent family members

Inter national Application No

PCT/GB 99/00064

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